

WHC-EP-0668 UC-600

Tank Characterization Report: Tank 241-C-109

B. C. Simpson G. L. Borshiem L. Jensen

Date Published September 1993

Prepared for the U.S. Department of Energy Office of Environmental Restoration and Waste Management



westingnouse
Hanford Company
P.O. Box 1970
Richland, Washington 99352

Hanford Operations and Engineering Contractor for the U.S. Department of Energy under Contract DE-AC06-87RL10930



This page intentionally left blank.



LEGAL DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, nor any of their contractors, subcontractors or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or any third party's use or the results of such use of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof or its contractors or subcontractors. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

This report has been reproduced from the best available copy. Available in paper copy and microfiche.

Available to the U.S. Department of Energy and its contractors from Office of Scientific and Technical Information P.O. Box 62 Oak Ridge, TN 37831 (615) 576-8401

Available to the public from the U.S. Department of Commerce National Technical Information Service 5285 Port Royal Road Springfield, VA 22161 (703) 487-4650

Printed in the United States of America

DISCLM-1.CHP (1-91)

THIS PAGE INTENTIONALLY LEFT BLANK

The property of the second second

Tank Characterization Data Report: Tank 241-C-109 Document Title: Prepared by: Brett C. Simpson / Advanced Engineer Analytical Evaluation and Reporting Prepared by: George L. Borsheim, Fellow Engineer Tank Waste Technology Applications Approved by: Harry Babad, Advisory Scientist Waste Tank Safety Programs 16 Kenl Approved by: Nicholas W. Kirch, Manager Tank Waste Technology Applications Approved by: Noonan, Manager Analytical Evaluation and Reporting Approved by: Robert J. Cash, Manager Ferrocyanide Safety Technology Approved by: John C. Fulton, Manager Waste Tank Safety Programs Approved by: rosson John G. Própson, Manager Characterization Program Approved by: Donald C. Board, Manager Quality Assurance Approved by: coll Muhammed N. Islam, Manager Waste Tank Safety Analysis

	Date Received: 9-22-93		INFO	RMATI	ON RE	LEAS	SE RE	QUEST	Reference: WHC-CM-3-4					
				Complete	for all Ty	pes of								
	[] Speech or Presentation				rence nical Repor			ber (include revision, volu EP-0668	me, etc.)					
	[] Full	y one		is or	•									
	[] Summa [] Abstr	' 7	יואי (אויד	[] Manual [] Brochure/Flier				None						
	[] Visue				tware/Database trolled Document Date Release Required									
	[] Speakers Bureau [] Other					9-30-93								
	[] Videotape Title Tank Chara	a Report:	241-C-109			<u> </u>	Unclassified Category UC-600	Impact Level 2						
	New or novel (pate If "Yes", has disc company?		mitted by		[] Yes er	propr	ietary d	eceived from others in conf ata, trade secrets, and/or Yes (Identify)	idence, such as inventions?					
	Copyrights? [X] If "Yes", has writ [] No [] Yes	been gran	nted?			marks? No []	Yes (Identify)							
·			<u> </u>	Complete f	or Speech	or Pres	entation	- Language						
# J	Title of Conference	ce or Meeting						Sponsoring						
-	Date(s) of Confere	ence or Meeting	City/St	ate		put	ol i shed?	edings be [] Yes al be handed	[] No					
FERNIZ MELTIN VISE Urgania	Title of Journal		<u> </u>			out	t?							
	Title of Jodinal													
Ì				CHECK	LIST FOR S	IGNATOR	IES							
	Review Required pe	r WHC-CM-3-4	<u>Yes</u>		Reviewer -	Signat	ure Indi	cates Approval						
	Classification/Unc		[]	[X] _	<u>Name</u>	(printe	<u>ed)</u>	<u>Signature</u>	<u>Date</u>					
	Patent - General C	Counsel	[X]	[] _	S. W. B O.G.C M	emo 2	<u>/4/93</u>							
	Legal - General Co	unsel	[X]		S. W. B O.G.C M									
	Applied Technology Controlled Informa	/Export			0.0.0	emo z	/ 4/ 33							
ł	International Prog WHC Program/Projec		[]	[X] _			 .	-1. A						
]	Communications	t	[X]		A. F. N	<u>oonan</u>		M/ Mhu	9/22/93					
1	RL Program/Project		[]	[X] _	1 14 6	 								
-	Publication Servic		[X]		J. M. C		- A A -	E) Hall for	5111 (
	Other Program/Proj		[X]		<u>C. F. F</u>	orbes	M.k	Coldbell for XI.	Oldfield 9/					
			[]	[X]	Th				<i>U</i>					
 	Information confor	ns to all applic	able requ	No No	ine above			certified to be correct. LEASE ADMINISTRATION APPROV	/AL STAMP					
	References Availab Audience	le to intended	[X]	[]	Stamp resolut	s requi	red befo	re release. Release is cor y comments.						
	Transmit to DOE-HQ Scientific and Tec Information	/Office of hnical	[X]	[]			A.	TEO LOS DE						
	Author/Requestor (Printed/Signatur	e)	Date			20							
	B. C. Simpson Intended Audience	Butten	-34 m	9/22/93	3		d	SE						
	[] Internal	[] Sponsor	Y1				S. S	9/20/13/						
	Responsible Manager		X] Exte					The state of the s						
				Date		···········								
1	A. F. Noonan	mulan	کسر ج	127/53	Date Ca	ncelled		Date Disepproved						

EXECUTIVE SUMMARY

Single-shell tank 241-C-109 is a Hanford Site Ferrocyanide Watch List tank that was most recently sampled in September 1992. Analyses of materials obtained from tank 241-C-109 were conducted to support the resolution of the ferrocyanide unreviewed safety question (USQ) and to support Hanford Federal Facility Agreement and Consent Order¹ (Tri-Party Agreement) Milestone M-10-00.

Analysis of core samples obtained from tank 241-C-109 strongly indicates that the fuel (e.g., ferrocyanide and organic) concentration in the tank waste will not support a propagating exothermic reaction. Analysis of the process history of the tank as well as studies of simulants provided valuable information about the physical and chemical condition of the waste. This information, in combination with the analysis of the tank waste, supports the conclusion that an exothermic reaction in tank 241-C-109 is not plausible. Therefore, the contents of tank 241-C-109 present no imminent threat to the workers at the Hanford Site, the public, or the environment from its ferrocyanide inventory. Because a propagating, exothermic reaction is not credible, the consequences of this accident scenario, as promulgated by the General Accounting Office, are not applicable.²

¹Ecology, EPA, and DOE, 1992, Hanford Federal Facility Agreement and Consent Order, 2 vols., Washington State Department of Ecology, U.S. Environmental Protection Agency, and U.S. Department of Energy, Olympia, Washington.

²Peach, J. D., 1990, "Consequences of Explosion of Hanford's Single-Shell Tank are Understated," (Letter B-241479 to C. M. Synar, Chairman of Environment, Energy, and Natural Resources Subcommittee, Committee on Government Operations, House of Representatives), GAO/RCED-91-34, General Accounting Office, Washington, D.C.

It is probable that tank 241-C-109 exceeds the 1,000 g-mol inventory criterion established for placement on the Ferrocyanide Watch List. However, extensive energetic analysis of this waste has determined a maximum exothermic value of -12.4 cal/g dry waste, and in most cases, no discernable exotherm was observed. This observation was further substantiated by total cyanide measurements of less than 1.5 dry weight percent. This exothermic measurement is substantially below the established level of concern, -75 cal/g ¹. In addition, an investigation of potential mechanisms to generate concentration levels of radionuclides high enough to be of concern (i.e., to cause in-tank self-heating) was performed. No credible mechanism was postulated that could initiate the formation of such concentration levels² in the tank.

Tank 241-C-109 waste is a complex material made up primarily of water and inert salts. The insoluble solids are a mixture of phosphates, sulfates, and hydroxides in combination with aluminum, calcium, iron, nickel, and uranium. Disodium nickel ferrocyanide and sodium cesium nickel ferrocyanide probably exist in the tank; however, there appears to have been significant degradation of this material since the waste was initially settled in the tank. Most of the ¹³⁷Cs precipitated during the scavenging campaign (1955 to 1957)³ appears to still remain in the tank in an insoluble form, probably bound with the remaining ferrocyanide. During the tank's service life, additional ¹³⁷Cs and

¹Jewett, J. R., 1992, "Energy Measurements for Disqualifying Waste Tanks from Watch Lists," Meeting Minutes, October 22, 1992, Westinghouse Hanford Company, Richland, Washington.

²Dickinson, D. R., J. M. McLaren, G. L. Borsheim, and M. D. Crippen, 1993, *Credibility of Drying Out Ferrocyanide Tank Waste Sludge by Hot Spots*, WHC-EP-0648, Westinghouse Hanford Company, Richland, Washington.

³Borsheim, G. L. and B. C. Simpson, 1991, *An Assessment of the Inventories of the Ferrocyanide Watchlist Tanks*, WHC-SD-WM-ER-133, Rev. O, Westinghouse Hanford Company, Richland, Washington.

⁹⁰Sr was deposited after the scavenging campaign. The most prevalent soluble analytes are primarily sodium, nitrate, and nitrite (refer to Table ES-1).

Comparisons of the calculated bulk inventories for various analytes of concern show that tank 241-C-109 is within established operating safety requirements for heat-load, organic content, and plutonium inventory.

Tank 241-C-109 is considered a sound, non-leaking tank.

	•
	Characterization
ı	cter:
	izati
	Summary
	for
	S

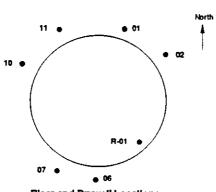
Estimated major analyte inventory	Water	Sodium	NO3	Uranium	NO ₂	Phosphorus	Iron	Calcium	Nickel	Aluminum	TOC			
Weight percent (Wt%) (wet solids)	36.5	8.1	4.0	1.1	4.0	1.7	2.5	1.8	2.5	7.6	0.26			
Estimated total waste Supernate: 18.7 Mg Wet solids: 284.6 Mg	mass in ta	nk 241-C-10	9: 303.3	Mg										
Estimated fission pro	duct invent	огу			<u> </u>	137 _{Cs}			⁹⁰ sr					
Bulk inventory, Ci (wet solids)						209,900 ci			264,600 Ci					
Heat generation, W						991 W			1,772 W					
Estimated plutonium/a	mericium ir	ventory	238 _{Pu}			239 _{Pu}			241 _{Am}					
Bulk inventory, Ci (wet solids)			0.012 Ci			233.5 Ci			91.1 Ci					
Bulk inventory, g (wet solids)	7.3E-04	¥ g		3,800 g		· · · · · · · · · · · · · · · · · · ·	26.5 g							
Analyte			Safety	issue crite	ria				Calculated/measured value					
Na ₂ NiFe(CN) ₆ (Wet sol	ids)	-	1,000	g-mot					6,800 g-mol					
AH (dry basis)			-75 cal/g						-12.4 cal/g					
239/240 _{Pu}	50 kg						3.8 kg							
Temperature	300 °F (149 °C)						78 °F (26 °C)							
Heat load 11.72 km				1.72 kw						2.76 kw				
Organic content (TOC, dry basis)				3.0 Wt% TOC (10% sodium acetate equivalent)						0.41 Wt%				

TOC = Total organic carbon.

S.

Figure ES-1. Tank 241-C-109 Summary Tank Data.

Tank: 241-C-109



Riser and Drywell Locations Tank 241-C-109

Tank Description

Type: Single Shell
Constructed: 1944
In-service: 04/48
Out of Service: 1976
Diameter: 75'
Usable Depth: 16'
Capacity: 530K gallons
Bottom shape: Dish
Hanford Coordinates:
43,003' North
48,327' West

Ventilation: Passive

Leak Detection System

Surface Level:

FIC Riser- None Manual Tape Riser- R-01 LOW Riser(s)- None

Number of External Drywells: 6 Number of Lateral Wells: None

Tank Status

Watch List: Ferrocyanide

Contents

Type: Non-Complexed Waste Total Waste: 66K gallons Supernate Volume: 4K gallons

Drainable Interstitial Liquid: OK gallons

Isolation Status

Date Interim Stabilized: 11/29/83 Date Interim Isolated: 12/15/82

Surface Level/Leak Status

Integrity Category: Sound

Manual Tape Surface Level: 18.00 Inches (11/23/92)

Last Photographed: 01/30/76

Temperature Status

Highest temperature during 1992:

88 deg F (09/30/92)

Comments:

Temperatures are stable.

Drywell Status

Comments:

Current drywell profiles were stable and consistent with established baseline profiles.

This page intentionally left blank.

CONTENTS

1.0	1.1	DUCTION
2.0	2.1	MPLING INFORMATION AND EVALUATION
3.0	CORE 3.1 3.2	SAMPLING
4.0	4.1	E PREPARATION/SAMPLE EXTRUSION 4-1 SAMPLE BREAKDOWN PROCEDURE 4-1 HOMOGENIZATION TESTS 4-2 SUBSEGMENT-LEVEL ANALYSES 4-3 4.3.1 Rheological and Physical Measurements 4-4 4.3.2 Subsegment Level Archive 4-4 4.3.3 Core Composite Level Analysis 4-5
5.0	5.1 5.2	TICAL RESULTS: TANK 241-C-109
		TANK 241-C-109 CORE SAMPLE RHEOLOGICAL/ PHYSICAL MEASUREMENTS
	5.4	5.3.4 Particle Size Measurement
		ATOMIC EMISSION SPECTROSCOPY

CONTENTS (Continued)

		5.4.2 Liquid Core Composite 5-26
		5.4.3 Core 47
		5.4.4 Core 48
		5.4.5 Core 49
	5.5	5.4.5 Core 49
	0.0	5.5.1 Ion Chromatography AssaysGeneral Comments 5-29
		5.5.2 Liquid Core Composite
		in the complete contraction of the contraction of t
		5.5.4 Core 48
		5.5.5 Core 49
	5.6	ANALYTICAL RESULTSRADIOCHEMISTRY 5-34
		5.6.1 Radiochemistry AssaysGeneral Comments 5-34
		5.6.2 Gamma Energy Analysis Results 5-34
		5.6.3 Total Alpha Analysis and Uranium Assav 5-35
		5.6.4 Total Beta 5-39
	5.7	ANALYTICAL RESULTS ENERGETICS 5-30
	3.,	5.6.4 Total Beta
		Sanning Calculation of Differential
		Scanning Calorimetry/Thermogravimetric
		Analysis Data
		5.7.2 General Comments on the Differential
		Scanning Calorimetry/Thermogravimetric
		Analysis Behavior of the Samples
	5.8	ANALYTICAL RESULTS - POTENTIAL WASTE CONSTITUENTS 5-46
		5.8.1 Mass Balances
		5 8 2 Suggested Components of Wasta Matrix
		5.8.3 Comparison to Theoretical Estimates
		5.8.3 Comparison to Theoretical Estimates and Simulant Studies
	5.9	DCDA_TVDE ANALYCIS. DATA VALIDATION/VEDICICATION DDGTGCG. F. F.
	3.3	E 0.1 Chemical Data
		5.9.1 Chemical Data
		5.9.2 Radiological Data
	T 1.17 P 1	DOCTATION OF ANALYTICAL DEGINERA
6.0	TNIF	RPRETATION OF ANALYTICAL RESULTS 6-1
	6.1	TANK 241-C-109 WASTE PROFILE
	6.2	REVIEW OF THE SUBSEGMENT ANALYTE PROFILES 6-2
		6.2.1 Tank Entrance/Exit Effects
		on Analyte Distribution 6-3
	6.3	6.2.1 Tank Entrance/Exit Effects on Analyte Distribution
		COMPARISONS WITH THE BORSHEIM/SIMPSON MODEL ESTIMATES 6-8
	0.4	CONTAKTORS WITH THE BOKSHEIM/SIMFSON MODEL ESTIMATES 0-8
7.0	OHANT	TITATIVE/STATISTICAL INTERDRETATION OF THE DATA
7.0	7.1	TITATIVE/STATISTICAL INTERPRETATION OF THE DATA
	7.1	INTRODUCTION
	7.2	MEAN CONCENTRATION ESTIMATES
	7.3	HOMOGENIZATION TEST
	7.4	HOMOGENIZATION TEST
	7.5	THE SPATIAL VARIANCE AND ANALYTICAL MEASUREMENT VARIANCE 7-9
	7.6	MULTIPLE COMPARISONS: CORE COMPOSITE
		SAMPLES AND SUBSEGMENT SAMPLES . 7_11
	7.7	SAMPLES AND SUBSEGMENT SAMPLES
		TANKS 241-C-109 AND 241-C-112
		7.7.1 Comparison of Mean Concentrations
		7.7.2 Companion of Variance
		7.7.2 Comparison of Variances $\dots \dots \dots$

CONTENTS (Continued)

	7.8 SUMMARY OF STATISTICAL INTERPRETATION		•	•	•	•	•		•	•	•	•		•	7-19
8.0	CONCLUSIONS AND RECOMMENDATIONS	•						•		•	•				8-1 8-2
9.0	REFERENCES	•	•	•	•	•		•	•		•	•	•	•	9-1
	APPENDIXES														
Α	CALCULATIONS		•			•	•	•			•			•	A-1
В	STATISTICAL INTERPRETATION	•	•	•		•				•	•		•		B-1
С	MISCELLANEOUS DATA	•	•	•		•			•		•		•	•	C-1
D	QUALITY ASSURANCE DOCUMENTATION SUMMARY .														D-1

LIST OF FIGURES

2-1	Typical Single-Shell Tank Diagram
2-2	In Farm Flowsheet
2-3	Solids and Liquid Wastes - Tank 241-C-109
2-4	Ferrocyanide Tank 3-Component Diagram
3-1	Tank 241-C-109 Riser Configuration
4-1	Typical Single-Shell Tank Segment Extrusion
5-1	Current Waste Level of Tank 241-C-109
5-2	Core 47 Measurements and Observations
5-3	Core 48 Measurements and Observations
5-4	Core 49 Measurements and Observations
5-5	Shear Stress Versus Shear Rate for 1:1 Diluted Sample 5-14
5-6	Viscosity Versus Shear Rate for 1:1 Diluted Sample 5-14
5-7	Core 47, Particle Size Number Density 5-16
5-8	Single-Shell Tank Core 47, Particle Size Volume Density
5-9	Core 48, Particle Size Number Density
5-10	Single-Shell Tank Core 48, Particle Size Volume Density
5-11	Core 49, Particle Size Number Density
5-12	Single-Shell Tank Core 49, Particle Size Volume Density
5-13	Settling Rate Data for Tank 241-C-109 Core 49, 1:1 Dilution
5-14	Settling Rate Data for Tank 241-C-109 Core 49, 3:1 Dilution
6-1	Elevation and Plan of Tank 241-C-109 6-4
6-2	Waste Profile of Tank 241-C-109 6-5

LIST OF TABLES

2-1	Expected Concentrations for Characteristic Analytes 2-10
2-2	Feed Solution Composition for In Farm 2 Flowsheet
2-3	Summary of In Farm 2 Simulant Characterization Data 2-11
2-4	Estimated Composition of Homogenized, Centrifuged, Ferrocyanide Sludge Simulant
2-5	Heats of Reaction of Various Simulants
3-1	Chain-of-Custody Summary
4-1	Subsegment-Level Analysis
5-1	Tank 241-C-109 Core Sample Description Summary 5-1
5-2	Tank 241-C-109 Core Sample Physical Characteristics Summary 5-1
5-3	Retrieval Program
5-4	Trace Analytes of Interest to Pretreatment
5-5	Core Composite Transuranics (Fusion Preparation) 5-5
5-6	Core Composite Uranium
5-7	Plutonium Concentration and Isotopic Distribution 5-5
5-8	HWVP Process Control Analytes of Concern
5-9	HWVP Regulatory Operation Analytes of Concern
5-10	Power-Law Model Parameters for Tank 241-C-109 Material 5-13
5-11	Turbulent Flow Model Calculations
5-12	Particle Size Distribution by Number: 89% < 2 μ m (all cores) 5-17
5-13	Particle Size Distribution by Volume: $100\% < 70\mu\text{m}$ (all cores) 5-17
5-14	Physical Properties Summary
5-15	Water Digestion Chemical Composite DataICP Average Values
5-16	Acid Digestion Chemical Composite DataICP Average Values
5-17	Fusion Digestion Chemical Composite DataICP Average Values

LIST OF TABLES (Continued)

5-18	Tank 241-C-109 Core 47 ICP Analyte Trending (Fusion Prep on Subsegments)
5-19	Tank 241-C-109 Core 48 ICP Analyte Trending (Fusion Prep on Subsegments)
5-20	Tank 241-C-109 Core 49 ICP Analyte Trending (Fusion Prep on Subsegments)
5-21	Anion AssaysComposite Data Results (Water Leach) 5-31
5-22	Tank 241-C-109 Core 47 IC Analyte Trending
5-23	Tank 241-C-109 Core 48 IC Analyte Trending
5-24	Tank 241-C-109 Core 49 IC Analyte Trending
5-25	Tank 241-C-109 Core 47 Misc. Analyte Trending 5-33
5-26	Tank 241-C-109 Core 48 Miscellaneous Analyte Trending 5-33
5-27	Tank 241-C-109 Core 49 Miscellaneous Analyte Trending 5-34
5-28	Core Composite Fission Products (Fusion Prep)
5-29	Liquid Core Composite Fission Products (Acid Prep) 5-36
5-30	Core Composite Uranium
5-31	Core Composite Trace Radionuclides
5-32	Plutonium Concentration and Isotopic Distribution 5-37
5-33	Core Composite Transuranics (fusion preparation) 5-38
5-34	Total Alpha Homogenization Test (μ Ci/g) (Acid Prep) 5-38
5-35	Tank 241-C-109 Core 47 Radionuclide Trending (Fusion Preparation)
5-36	Tank 241-C-109 Core 48 Radionuclide Trending (Fusion Preparation)
5-37	Tank 241-C-109 Core 49 Radionuclide Trending (Fusion Preparation)
5-38	Thermogravimetric Analysis Results from Tank 241-C-109

LIST OF TABLES (Continued)

5-39	Differential Scanning Calorimetry Energetics Results from Tank 241-C-109
5-40	Tank 241-C-109 Core 47 Energetic Comparison
5-41	Tank 241-C-109 Core 48 Energetic Comparison 5-44
5-42	Tank 241-C-109 Core 49 Energetic Comparison 5-44
5-43	Tank 241-C-109 Core 47 Energetics Trending 5-45
5-44	Tank 241-C-109 Core 48 Energetics Trending 5-45
5-45	Tank 241-C-109 Core 49 Energetics Trending 5-45
5-46	Adjusted Mass Balance: Core 47 Composite
5-47	Adjusted Mass Balance: Core 48 Composite
5-48	Adjusted Mass Balance: Core 49 Composite
5-49	Probable Solids in the Waste Matrix
5-50	Tank 241-C-109 Comparison of Waste Material with Simulants for Selected Analytes
6-1	Energetics Related Analyte Values 6-6
6-2	Fission Product Inventory 6-6
6-3	Plutonium/Americium Inventory 6-7
6-4	ICP Major Cation Inventory (From Fusion Preparation Results) 6-7
6-5	Comparisons of Initial and Revised Borsheim/Simpson Model Estimates with Values Calculated from Analytical Results 6-7
7-1	Core Composite Data (Units $\mu g/g$ Except Radionuclides $\mu Ci/g$) 7-2
7-2	Subsegment Data (Unites $\mu g/g$ Except Radionuclides $\mu Ci/g$) 7-3
7-3	Homogenization Test Data (Units $\mu g/g$ Except Radionuclides $\mu Ci/g$)
7-4	Concentration Estimate Statistics (Units $\mu g/g$ Except Radionuclide $\mu Ci/g$)
7-5	Concentration Estimates Statistics, Drainable Liquid. (Units $\mu q/q$)

LIST OF TABLES (Continued)

7-6	Homogenization Test Statistical Results
7-7	Comparison of Simulated Core Composite with the Core Composite 7-10
7-8	95 Percent Confidence Interval on $\sigma^2(A)$, Analytical Error Variance
7-9	95 Percent Confidence Interval on $\sigma^2(S)$, Spatial Variance
7-10	Tukey's HSD Multiple Comparisons, Core Composite Data (Units μ g/g Except Radionuclides μ Ci/g)
7-11	Subsegment Assignment for Tukey's HSD
7-12	Tukey's HSD Multiple Comparisons, Subsegment Data (Units $\mu g/g$ Except Radionuclides $\mu Ci/g$)
7-13	Core Composite Assignment for Tank Comparison
7-14	Comparison of Analytical Error Variances, Tanks 241-C-109 and 241-C-112
7-15	Comparison of Spatial Variances, Tanks 241-C-109 and 241-C-112
7-16	Comparison of Percent Variance, 241-C-109 and 241-C-112
7-17	Relative Standard Deviations from Tanks C-112 and C-109
8-1	Comparison of Tank 241-C-109 Analyte Values to Safety Issue Criteria

LIST OF ACRONYMS

1C	first decontamination cycle
AA	atomic absorption
AEA	alpha energy analysis
ANOVA	analysis of variance
ASC	adiabatic scanning calorimetry
CI	confidence interval
CST	core sampling truck
CW	decladding/coating waste
DLC	drainable liquid composites
DSC	differential scanning calorimetry
EB	evaporator bottoms
EPA	U.S. Environmental Protection Agency
GAO	General Accounting Office
GEA	gamma energy analysis
HASM	Hanford Analytical Services Management
HDW-EIS	Final Environmental Impact Statement, Disposal of
,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	Hanford Defense High-Level, Transuranic and Tank
•	Wastes
HS	hot semiworks
HWVP	Hanford Waste Vitrification Plant
IC	ion chromatography
ĪCP	inductively coupled plasma - atomic emission
101	spectroscopy
NCAW	neutralized current acid waste
PNL	Pacific Northwest Laboratory
RPD	relative percent difference
SST	single-shell tank
TC	total carbon
TGA	thermogravimetric analysis
TIC	total inorganic carbon
TOC	total organic carbon
TOD	total oxygen demand
Tri-Party	total oxygen demand
_	Hanford Federal Facility Agreement and Consent Order
Agreement TRU	transuranic
UR .	uranium recovery
USQ	unreviewed safety question

This page intentionally left blank.

TANK CHARACTERIZATION DATA REPORT: TANK 241-C-109

1.0 INTRODUCTION

Analysis was conducted on materials obtained from tank 241-C-109 to support the closure of the ferrocyanide unreviewed safety question (USQ). Obtaining measurements that determine overall waste energetics is a key step in closing the ferrocyanide USQ and safety issue. In addition, several of the analytes contributing to the energetic properties of the waste need to be measured as a function of position (e.g., total cyanide and nitrate/nitrite present, water content, and the distribution and inventory of ¹³⁷Cs and ⁹⁰Sr in the tank). Other objectives that these measurements and inventory estimates support are as follows.

- Complete Hanford Federal Facility Agreement and Consent Order (Tri-Party Agreement) Milestone M-10-00 (Ecology et al. 1992) to sample and analyze two cores from each tank.
- Obtain estimates of both the concentration and total quantity of key analytes relating to other safety issues, such as organics and radionuclides.
- Provide input to risk assessment-based disposal decisions for the waste.
- Implement physical property measurements, such as rheology, bulk density, and particle size. These measurements are necessary for the design and fabrication of retrieval, pretreatment, and final waste disposal systems.

1.1 PURPOSE

This report summarizes the available information regarding the waste in tank 241-C-109, and arranges this information in a useful format for data users in various internal and external organizations.

1.2 SCOPE

This report presents a broad background of preliminary information that was available prior to core sampling, and which initially guided the development of the sampling and analysis program. This material includes historical information about the ferrocyanide-scavenging program, transfer records, observations from in-tank photographs, and inferences from waste simulant studies. The results of tank 241-C-109 core sample analyses are summarized and presented, along with a statistical interpretation of the data. The information obtained from historical sources and synthetic waste studies are compared with the actual waste measurements in this report.

This page intentionally left blank.

2.0 PRESAMPLING INFORMATION AND EVALUATION

2.1 BACKGROUND

Radioactive wastes from defense operations have accumulated at the Hanford Site in underground waste tanks since the 1940's. During the 1950's, additional tank storage space was required to support the United States defense mission. To obtain additional tank storage volume within a short period of time and to minimize construction of additional storage tanks, Hanford Site scientists developed a process to scavenge radiocesium from tank waste liquids (Sloat 1954, Abrams 1956). Ferrocyanide compounds were used in a carrier-precipitation process to scavenge ¹³⁷Cs and other soluble radionuclides from the Hanford Site waste tanks. This treatment was used on U Plant waste effluent, bismuth phosphate first decontamination cycle waste, and selected wastes that had been previously discharged to the tanks. Some of these wastes had been processed through the 242-B Evaporator prior to scavenging. The radionuclides settled in the waste tanks and the supernate was discharged to the cribs and trenches. As a result of this process, occupied waste volume in the waste tanks was greatly reduced, while minimizing the amount of long-lived radionuclides discharged to the ground.

In implementing this process, approximately 140 metric tons of ferrocyanide [as $Fe(CN)_6^4$] were added to the tanks. The bulk of the ferrocyanide material is believed to remain in 18 to 24 single-shell tanks (SSTs). Ferrocyanide is a stable complex of iron(II) ion and cyanide, whose compounds are considered nontoxic because they do not appreciably dissociate in aqueous solutions (Burger 1989). However, recent laboratory data indicates that highly alkaline solutions can degrade ferrocyanide salts (Babad 1993a,b). In the presence of oxidizing materials such as nitrates and/or nitrites, ferrocyanide compounds can undergo uncontrolled exothermic reactions in the laboratory by heating them to high temperatures (above 280 °C [540 °F]). Because the scavenging process involved precipitating ferrocyanides from solutions containing nitrate and nitrite, the potential for a reactive mixture of ferrocyanides and nitrates/nitrites in the SSTs must be evaluated.

2.1.1 Tank 241-C-109 History

Groups of waste tanks that were physically located together and built at the same time are called tank farms at the Hanford Site. The original tank farms (B, C, T, U) were built from 1943 to 1944. Each tank has a diameter of 22.9 m (75 ft), an operating depth of 5.2 m (17 ft), and a nominal capacity of 2 million liters (530,000 gal). The basic design of a typical SST is shown in Figure 2-1. The tank was constructed of reinforced concrete with a mild steel liner covering its bottom and sides. The top of the tank is a concrete dome. Tanks such as 241-C-109 were covered by at least 1.8 m (6 ft) of soil for shielding purposes (Anderson 1990). Tank 241-C-109 was placed into service in 1946.

The tanks in the tank farms were connected in groups of three or four and overflowed from one to another (known as a cascade). Tank 241-C-109 is the last tank in a cascade that includes 241-C-107 and 241-C-108.

Dome Elevation Bench Mark Camera Observation Port Temperature Thermocouple Sludge Level HEPA Filter Conductivity Assembly Detector Probe Liquid Observation Well Drywell Reinforced Concrete Tank Steel Liner Supernatant Saltcake and/or Sludge

Schematic - Not to Scale

28905008.2

Figure 2-1. Typical Single-Shell Tank Diagram.

NOTE: Tank 241-C-109 has a dished bottom.

Cascades served several functions in Hanford Site waste management operations. By cascading tanks, fewer connections needed to be made during waste disposal. Consequently, all three tanks were usable without having to connect the active waste transfer line directly to each individual tank. This handling method reduced the likelihood for personnel exposure to the waste and diminished the chances for a loss of tank integrity because of overfilling. Another benefit of the cascades was clarification of the wastes. When used in this manner, most of the solids in the waste slurries routed to the tanks settled in the first tank (241-C-107), and the clarified liquids cascaded on to the other tanks in the series (241-C-108 and 241-C-109). Supernate from the final tank in a cascade series was sometimes routed to a disposal trench. In this way clarification reduced the potential amount of radiological contamination to the environment.

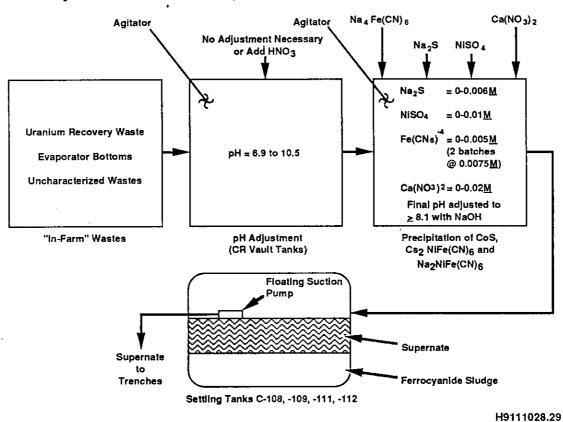
The first type of waste that tank 241-C-109 received and stored was first decontamination cycle waste from the bismuth phosphate process (1946 to 1952). This waste would be comparatively high in bismuth, phosphate, and aluminum because aluminum decladding waste was combined with it. The supernatant waste was transferred to tank 241-B-106 in 1952, leaving a 38,000-L (10,000-gal) heel, probably mostly solids. The tank was refilled with unscavenged uranium recovery (UR) waste in 1953 (Anderson 1990). The UR waste solids were comparatively high in uranium and iron, and low in bismuth and aluminum. The available records do not show whether these wastes were added directly to the tank or through the cascade overflow line from tank 241-C-108. Neither of these waste types had any significant fuel content or heat-generating radionuclides (137Cs or 90Sr), that could contribute to the exothermic potential posed by the ferrocyanide wastes.

Beginning in May 1955, unscavenged UR waste already stored in 200 East Area underground tanks at the Hanford Site was routed to the 244-CR vault for scavenging (refer to Figure 2-2). The 244-CR vault facility contained stainless steel tanks with chemical addition, agitation, and sampling capabilities. The pH was adjusted with HNO₃ and/or NaOH to pH 9.3 ± 0.7, and Fe(CN)₆⁴ and Ni⁺² ion was added (generally to 0.005 M each) to precipitate ¹³⁷Cs. If laboratory analysis of the feed tank indicated additional ⁹⁰Sr decontamination was necessary, calcium nitrate was also added (Sloat 1955). There was also an effort to scavenge ⁶⁰Co with Na₂S. From late 1955 until 1958, tank 241-C-109 was used for settling scavenged ferrocyanide waste. During ferrocyanide-scavenging operations, waste was not cascaded through the tank 241-C-107, -108, -109 series. Tank 241-C-109 received the waste slurry in direct transfers from the process vessel (General Electric 1958). The scavenged waste was settled, sampled, and decanted to a crib. The settling tanks for this In Farm scavenged waste were tanks 241-C-108, 241-C-109, 241-C-111, and 241-C-112.

The In Farm precipitate comprises approximately 20 to 25 percent of the total ferrocyanide material in the Hanford Site tank farms. This material is expected to possess a much higher ferrocyanide concentration content than the more prevalent U Plant material (70 percent of the total ferrocyanide material). Analytes that differentiate ferrocyanide waste from other wastes are elevated levels of nickel, calcium, and ¹³⁷Cs. Over time, additional gravity settling may have compressed the waste layers, increasing the

Figure 2-2. In Farm Flowsheet.

Ferrocyanide Production by "In-Farm" Flowsheet



concentration of some of these analytes. However, the interactive effects of radiation and high pH conditions from later waste additions on the waste matrix is largely unknown. Exposure of the waste to these conditions is believed to have degraded the ferrocyanide. However, laboratory results confirming that hypothesis are still pending (Lilga et al. 1992; Babad et al. 1993a,b).

The first transfer of scavenged waste for settling was in the fourth quarter of 1955. In Farm scavenging was completed in December 1957 (General Electric 1958). The inventory of solids in tank 241-C-109 at the end of the ferrocyanide-scavenging program, as calculated by the Borsheim-Simpson (1991) model, was 413,000 L (109,000 gal) with essentially no free supernate. A revised calculation using more appropriate solid formation values (Appendix A) gives a total inventory of 413,000 L (109,000 gal) and a solid inventory of 220,000 L (58,000 gal). The scavenging record (General Electric 1958) gives the overall tank level as 0.89 m (2 ft 11 in.) (413,000 L [109,000 gal]). A History of the 200 Area Tank Farms (Anderson 1990) reports a total volume of 424,000 L (112,000 gal), but lists 341,000 L (90,000 gal) of that inventory as solids (the reading previous to this was 193,000 L [51,000 gal]). The waste inventory values believed to be most representative of the solids level (and overall waste inventory) in this timeframe range between 51,000 and 58,000 gal. The wide variation in the waste levels between sources is not reassuring and contributes to significant uncertainty regarding tank inventory calculations.

After the end of scavenging in early 1958, tank 241-C-109 remained in active service. However, the tank had relatively limited activity from 1958 to the end of its service life in 1980. In the third and fourth quarters of 1959, a total of 1.57 M L (415,000 gal) of highly alkaline cladding waste and evaporator bottoms (wastes known to contain substantial amounts of solids) were added to the tank, but the reported solids inventory (341,000 L [90,000 gal]) did not change (Anderson 1990). From the known information, it seems likely there would be an increase of solids and that a transcription error may have occurred. Cladding waste solids would have settled on top of the ferrocyanide sludge already present.

Several small transfers with relatively high concentrations of 90 Sr occurred after 1958. In 1962, 519,000 L (137,000 gal) of liquid was transferred to the BY Farm. Waste from the strontium semiworks/hot semiworks was then added at different times to the tank, increasing the total volume listed to 2.02 M L (535,000 gal) at the end of 1964 (the reported solids inventory was still 341,000 L [90,000 gal]). The listed volumes for the fourth quarter report in 1966 are a total volume of 2.09 M L (552,000 gal), with a solids volume of 299,000 L (79,000 gal) (Anderson 1990). While this solids level measurement was the second taken since additional waste was added to the tank following the last scavenging pumpout in 1958, it was the first to use a new electrode to perform the overall volume measurement.

The reported waste volume remained essentially unchanged (between 2.01 and 2.04 M L [543,000 and 552,000 gal]) until a receipt of 72,000 L (19,000 gal) from tank 241-C-203, and a transfer of 1.50 M L (397,000 gal) to tank 241-C-104 occurred in the first quarter of 1970. This transfer left a heel of at least 609,000 L (161,000 gal). A floating suction pump transfer would not have transferred any solids because the maximum reported solids

level was 413,000 L (109,000 gal) and it was probably lower than that. In addition, there was no mixing equipment in tank 241-C-109 to move the settled ferrocyanide solids into the overlying solids layer. In the second quarter of 1970, an additional transfer of 1.42 M L (375,000 gal) from tank 241-C-110 was received. Between 1970 and 1975, the reported solids volume fluctuated widely between 401,000 and 235,000 L (106,000 and 62,000 gal), and the total volume reported decreased from 2.06 M L to 235,000 L (543,000 to 62,000 gal) (Anderson 1990).

Some solids may have been transferred, as the reported tank solids volume decreased from 485,000 L (90,000 gal) to 299,000 L (79,000 gal). However, the solids transferred would have been those that settled on top of the ferrocyanide solids (i.e., cladding waste/evaporator bottoms solids; ferrocyanide waste levels are at ~58,000 gal). The wide fluctuation makes it difficult to derive any firm conclusions regarding the stratification in the tank. Overall sludge volume in the tank may have decreased somewhat between 1958 and 1975 with further settling and compaction from the weight of overlying solids. Although, the amount of sludge added since the end of the scavenging campaign is not easily quantifiable, it is likely that the measurements are biased high. Floating suction pumps do not transfer solids readily, and the movement of 76,000 to 152,000 L (20,000 to 40,000 gal) of solid seems unlikely. With the large amounts of concentrated wastes in this tank, there is the possibility that relatively unsaturated supernatants that were transferred into the tank later in its service life redissolved significant amounts of waste and distributed the material elsewhere in the tank farms. The final solids measurement prior to the end of active service (1980) and the present tank surveillance measurement (1983) are identical, 235,000 L (62,000 gal), and not much above the estimated ferrocyanide waste level (220,000 L [~58,000 gal]). Appendix A has the results of a model that represents the inventory changes in the tanks with various initial conditions and solids formation valves after scavenging operations were completed. Therefore, it is estimated that an additional 15,000 L (4,000 gal) of solids is remaining from the transfers into the tank on top of the ferrocyanide during its active service.

The last of the major waste types was aluminum cladding waste. These materials would be high in aluminum and silica, with a very high pH (>1.0 M NaOH; pH \geq 14). However, the solids volume contribution to the tank is unknown because the majority of the solids would be deposited in the first tank to receive the wastes, which was not tank 241-C-109. The high pH of this waste is considered a significant factor affecting the state of the waste matrix. Other wastes had discernable impacts on the bulk characteristics of the tank contents as well. The strontium semiworks waste had a small volume of waste added, but would have a very high 90 Sr content because it included strontium recovery and purification waste losses. The B Plant ion-exchange waste was primarily liquid and was not expected to contribute significantly to the solids in the tank.

2.1.2 Unreviewed Safety Question Declaration

Efforts have been underway since the mid-1980's to evaluate the potential of a ferrocyanide combustion reaction in Hanford Site SSTs (Burger 1989; Burger and Scheele 1990; Burger 1984). In 1987, the *Final Environmental*

Impact Statement, Disposal of Hanford Defense High Level, Transuranic and Tank Wastes, hereinafter referred to as the HDW-EIS (DOE 1987), was issued. In the HDW-EIS, it was projected that the bounding "worst-case" accident in a ferrocyanide tank would be an explosion resulting in a subsequent short-term radiation dose to the public of 200 mrem.

A later General Accounting Office (GAO) study postulated greater worst-case accident consequences, with independently calculated doses one to two orders of magnitude greater than the HDW-EIS (Peach 1990). In September 1990, a special Hanford Site Ferrocyanide Task Team was commissioned to address all issues involving the ferrocyanide tanks, including the consequences of a potential accident. On October 9, 1990, the Secretary of Energy announced that a supplemental environmental impact statement would be prepared containing an updated analysis of safety issues for the Hanford Site SSTs, including a hypothetical ferrocyanide explosion. In October 1990, the ferrocyanide issue was also declared an USQ because the consequences of the accident scenario (as calculated by the GAO) were outside the bounds of the current safety analyses for SSTs. Furthermore, additional monitoring of tanks with designated USQs was mandated by Public Law 101-510 (1990).

Using a computer model output (Jungfleisch 1984), process knowledge, and transfer records, 24 waste tanks have been identified at the Hanford Site as potentially containing 1,000 g-mol (465 lb) or more of ferrocyanide as the Fe(CN)₆⁻⁴ ion. On further investigation, six of these tanks are believed to have received less than 1,000 g-mol of ferrocyanide sludge and are therefore candidates for removal from the Watch List (Cash 1993). Tank 241-C-109 is on the Ferrocyanide Watch List because it was a known process tank during the ferrocyanide-scavenging campaigns.

2.2 EXPECTED TANK CONTENTS/CONDITIONS

Process knowledge obtained from historical records and waste simulants produced from the scavenging process flowsheets can be used to predict the major constituents and some general physical properties of the waste matrix in the waste tanks. Initially, the differences between the U Plant and In Farm ferrocyanide sludges were not fully appreciated. However, further investigation of the simulants showed that the In Farm process would be expected to precipitate approximately 1.0 to 1.3 vol% solids, and thus the sludge would have been deposited in the receiver tanks in layers approximately 3.6 to 6.1 cm (1.4 to 2.4 in.) thick. This is much less than the 4.25 vol% and 15- to 20-cm (6- to 8-in.) layers expected from the U Plant material. addition, there are some batches of waste that were scavenged and settled in tank 241-C-109 without ferrocyanide addition (scavenged for residual ^{60}Co and ^{90}Sr). These materials may have some superficial similarity to the ferrocyanide scavenged waste, however, they are expected to have no fuel content and thus, no exothermic potential (the S^2 used in the 60 Co scavenging process is believed to have been converted to SO_4^{2}). The In Farm scavenged ferrocyanide tanks (such as tank 241-C-109) are expected to contain relatively soft sludge, which can be push-mode sampled. This expectation was supported by inspection of in-tank photographs. The other waste solids that were added to the tank after the scavenging campaign are also expected to be soft. During its operating history, tank 241-C-109 was never subject to any of the

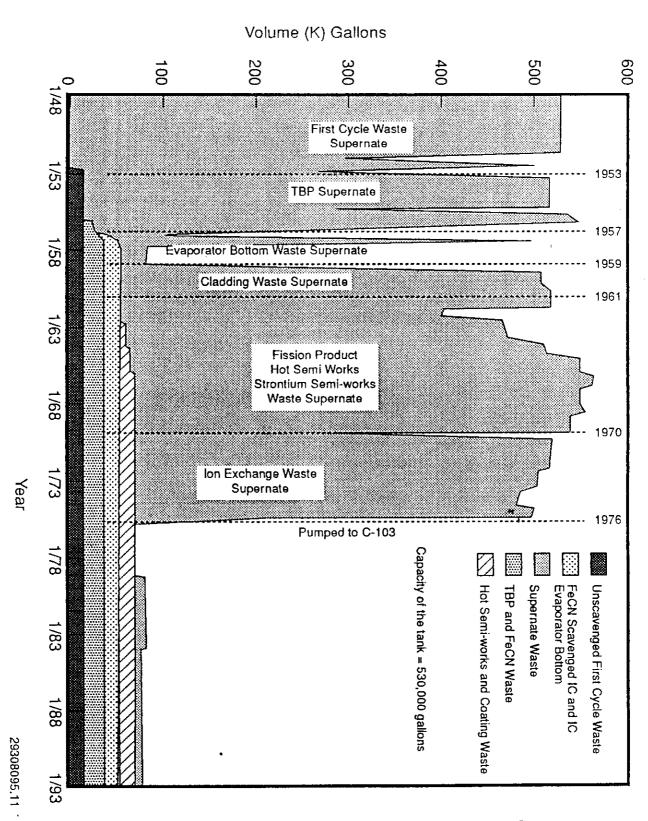


Figure 2-3. Solids and Liquid Wastes - Tank 241-C-109.

various in-tank solidification processes; consequently, there was no formation of hard salt cake on top of the sludge (as there was in the BY Tank Farm).

The most recent waste inventory report for tank 241-C-109 shows 235,000 L (62,000 gal) of solid waste with an estimated 15,000 L (4,000 gal) of drainable liquids (Hanlon 1992). These figures translate to a waste depth of 79.9 cm (31.5 in.) at the tank centerline. The tank was interim-stabilized in November 1983, and is considered sound. Tank Farm Operations has installed a second thermocouple tree in tank 241-C-109, and the readings between the two thermocouple trees on opposite sides of the tank are consistent. The current waste temperature in tank 241-C-109 is $\sim\!27$ °C (80 °F), and the estimated heat load in the tank is less than 2.93 kW (10,000 Btu/hr). Tank 241-C-109 is considered to have one of the highest ferrocyanide concentrations of all the ferrocyanide Watch List SSTs (Borsheim and Simpson 1991).

In summary, various nickel ferrocyanide complexes (primarily disodium) are expected to be mixed with an interstitial solution containing sodium nitrate and nitrite. Cesium-137 is expected to be present as a mixed salt (probably as NaCsNiFe(CN)₆); strontium-90 may be in several potential compounds; phosphate, sulfate, or carbonate. Both of these radionuclides have decayed through slightly more than one half-life, and therefore are not as abundant as when the scavenging waste was originally deposited. Other fission products with relatively short half-lives (such as ⁶⁰Co and ¹⁰⁶Ru) are not expected to be in abundance, especially with the limited number of waste additions for this tank. Hydrated transition metal oxides/hydroxides (including small amounts of transuranics) are also expected because of alkaline conditions. Other ions expected to be present are potassium, calcium, aluminum, and uranium. The supernate and interstitial liquid are expected to contain large amounts of sodium, nitrate, and nitrite ions. Table 2-1 presents some typical concentrations for identifier analytes in the waste streams disposed of in tank 241-C-109.

2.3 ANALYTICAL RESULTS FROM SIMULANT STUDIES

Physical and chemical measurements performed on simulants of ferrocyanide tank waste provide additional information and perspective regarding the condition and properties of the waste in tank 241-C-109.

2.3.1 Simulant Formulation: In Farm 2 Flowsheet Material

The In Farm 2 flowsheet material is considered to be an energetically conservative but reasonably close physical and chemical analogue of some of the ferrocyanide precipitate in tank 241-C-109 as it was deposited in the tank during the scavenging campaign. However, scavenging of evaporated cladding and first-cycle wastes is expected to produce noticeable differences from the uranium-recovered, scavenged TBP waste. In contrast to the results from 241-C-112, close agreement between the simulant properties and the waste measurements (physical and chemical analytes) was not expected. The In Farm 2 flowsheet materials were prepared according to the following instructions (Jeppson and Wong 1993). The feed solution composition is listed in Table 2-2. Deionized water was used for feed solution and chemical addition makeup.

Table 2-1. Expected Concentrations for Characteristic Analytes (Schneider 1951; Jeppson and Wong 1993).

•	• •	
Waste type	Identifier analytes	Typical process stream concentration (μg/g wet solids)
1 st Decontamination cycle (1C)	Bi	7,100
	F	9,600
	U	620
	Si	2,400
Unscavenged uranium recovery (UR)	U	1,700 - 32,500
Ferrocyanide (FeCN)	Ni	18,700
	Ca	30,000
	CN	91,000 - 113,000
Decladding waste (CW)	ΑΊ	72,200 - 96,200
	U	340 - 450
	Si	2,600 - 3,500
Hot semiworks (HS)	⁹⁰ Sr	No Data - expected elevated ⁹⁰ Sr levels

Table 2-2. Feed Solution Composition for In Farm 2 Flowsheet.

Component	Concentration
Sodium nitrate (NaNO ₃)	3.75 <u>M</u>
Cesium nitrate (CsNO ₃)	0.00025 <u>M</u>
Sodium nitrite (NaNO ₂)	1.25 <u>M</u>
Sodium sulfate (Na ₂ SO ₄)	0.17 <u>M</u>
Sodium phosphate (Na ₃ PO ₄)	0.16 <u>M</u>

The product sludge was the precipitate produced when performing the following steps. This procedure mimicked the actual In Farm 2 process that is illustrated in Figure 2-2. The feed solution was heated to 40 °C and the pH adjusted to 9.1 \pm 0.5. The sodium ferrocyanide was then added to the solution, followed by nickel sulfate. The simulant solution was agitated for 1 hour, then struck with calcium nitrate. After the addition of calcium nitrate, the solution was agitated for another hour and allowed to settle. The settling was done for eight days and the supernate was decanted. The remaining sludge was centrifuged at 2,100 g for 14 hours and 1,820 g for 7 days in an attempt to simulate 3.6 and 30 gravity-years of settling respectively (Jeppson and Wong 1993). Selected physical properties for the two settled centrifuged sludges are presented in Table 2-3. Table 2-4 presents an estimate of the chemical composition of the In Farm 2 simulant.

2.3.2 Simulant Physical Characteristics (see Jeppson and Wong 1993)

Table 2-3. Summary of In Farm 2 Simulant Characterization Data.

Centrifugation	Property	In Farm 2 sludge
30 gravity/yr	Water content, sludge	51 wt%
	pH, supernatant	9.42
	Bulk density, sludge	1.39 g/mL
3.6 gravity/yr	Bulk density, supernate	1.27 g/mL
	Particle density (dried sludge)	2.38 g/mL
	Particle size distribution, (by number)	97% < 2 μ m Median diameter*: 0.76, 0.76 μ m Acquisition Range: 0.5-150 μ m
	Particle size distribution, (by volume)	100% < 110 μm Median diameter*: 14.3, 16.8 μm Acquisition range: 0.5-150 μm
	Hydraulic conductivity (permeability)	4.0 x 10 ⁻⁷ cm/s
	Total porosity	67.9%
	Thermal conductivity	1.82 W/m•K @ 39 °C 2.16 W/m•K @ 56 °C 2.82 W/m•K @ 68 °C 2.04 W/m•K @ 72 °C**

^{*}Two separate measurements.

^{**}Jeppson and Wong (1993) noted an anomalous data point, but were unable to explain the inconsistency of the observation.

2.3.3 Simulant Chemical Composition (see Jeppson and Wong 1993)

Table 2-4.	Estimated Composition of Homogenize	d, Centrifuged,
	Ferrocyanide Sludge Simulant.	

Sludge component	Average In Farm 2 weight fraction
Disodium mononickel ferrocyanide: Na ₂ NiFe(CN) ₆	0.101
Sodium nitrate: NaNO ₃	0.174
Sodium nitrite: NaNO ₂	0.051
Sodium hydrogen phosphate: Na ₂ HPO ₄	0.016
Sodium sulfate: Na ₂ SO ₄	0.015
Calcium phosphate: Ca ₃ (PO ₄) ₂	0.072
Water	0.51
Percent mass balance subtotal	94.0
Percent unknownlikely includes $Fe_4(Fe(CN)_6)_3$, $Fe(OH)_3$, $Ni(OH)_2$, and other trace compounds from impurities.	6.0

2.3.4 Energetics Behavior of Ferrocyanide Sludge Simulant

Available chemical process information indicates that there were three significantly different types of ferrocyanide waste (Sloat 1954; Schmidt and Stedwell 1954). Nonradioactive waste simulants have been developed and tested using this information. In Farm ferrocyanide waste, accounting for 20 to 25 percent of the total ferrocyanide waste, was formed from treating waste that was already stored in the tanks. The waste in tank 241-C-109 was produced using the In Farm process. Most of this waste had less inert solids in the waste stream; therefore, it is believed to have been more concentrated in ferrocyanide than other scavenged wastes. In Farm simulants exhibit propagating exothermic activity when examined by differential and adiabatic scanning calorimetry (DSC and ASC) (Cady 1992; Fauske 1992).

Estimates of tank waste reactivity, which were developed after the ferrocyanide USQ was declared, were based on thermodynamic estimates (Colby and Crippen 1991). Several chemical reaction pathways were evaluated and heats of reaction were determined for each possible reaction from the published heats of formation of the reactants and the products. For the purpose of developing these estimates, the condition of the reactants is dry solid reagents at standard temperature and pressure in a stoichiometric ratio. The theoretical heats of reaction ranged in value from $\Delta H = -9.6 \text{ kJ/g}$ to $\Delta H = +19.7 \text{ kJ/g}$ of $Na_2NiFe(CN)_6$, and are listed below with their corresponding chemical reactions.

- (1) $Na_2NiFe(CN)_6 + 54NaNO_3 + 22H_2O ----> 6Na_2CO_3 + FeO + NiO + 60NO_2 + 44NaOH$ $<math>\Delta H = +19.7 \text{ kJ/g of } Na_2NiFe(CN)_6$
- (2) $Na_2NiFe(CN)_6 + 14NaNO_3 + 2H_2O ----> 6Na_2CO_3 + FeO + NiO + 20NO + 4NaOH$ $<math>\Delta H = -0.7 \text{ kJ/g of } Na_2NiFe(CN)_6$

At temperatures below 1700 °C (3100 °F), the carbonate product is thermodynamically favorable and should predominate (Scheele et al. 1991). Note that considerably lower energy releases are obtained if the reaction is incomplete or if NO or NO_2 is formed rather than N_2 or N_2O . A three-component diagram illustrating the exothermic potential of various mixtures of ferrocyanide, nitrate, and inerts is presented in Figure 2-4. Further detail regarding the thermodynamic estimates of these mixtures is presented in Colby and Crippen (1991).

The waste simulants prepared using the In Farm and U Plant process flowsheets were tested for chemical activity (Fauske 1992). Chemical and physical analyses of the In Farm and U Plant waste simulants show that they contain an average of 51 and 66 wt% water, respectively, after centrifugation. The centrifugation was done to represent 30 gravity-years of compaction that may have occurred during storage. This amount of water in the waste matrix presents a tremendous heat sink that must be overcome before any reactions can become self-sustaining. During the DSC examinations, the samples exhibited large endotherms between room temperature and 150 °C (Jeppson and Wong 1993). Results from thermogravimetric analyses being run at the same time showed a large loss of mass (i.e., evaporation of water) in this same temperature range; thus, reactions were only able to occur in dry or nearly dry sample material (Cady 1992). Average ferrocyanide content of the In Farm 2 waste simulants is approximately 10.1 wet wt% (20.6 wt% dry). Table 2-5 presents the ΔH found for some simulant materials.

Table 2-5. Heats of Reaction of Various Simulants (Fauske 1992).

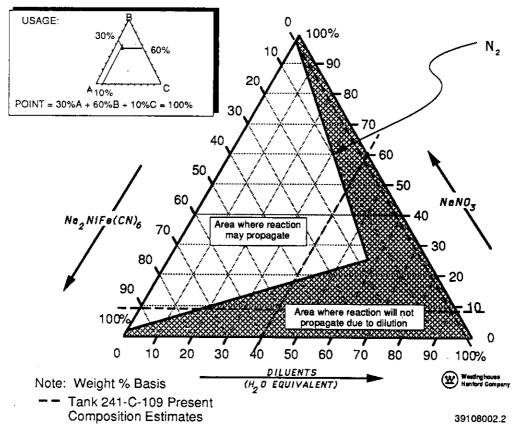
Material	ΔΗ (From adiabatic calorimetry)	Wt% Ferrocyanide (dry) [Na ₂ NiFe(CN) ₆]	Calculated ΔH per gram Na ₂ NiFe(CN) ₆	
U Plant 1 simulant	-0.17 kJ/g of dry material	4.3	-3.95 kJ/g	
U Plant 2 simulant (Bottom fraction)	-0.34 kJ/g of dry material	8.6	-3.95 kJ/g	
In Farm 1 simulant (Bottom fraction)	-1.20 kJ/g of dry material	25.5	-4.71 kJ/g	

The onset temperatures for propagating reactions to take place in the simulants range from 244 °C to 278 °C (471 to 532 °F). However, Arrhenius-type reactions may occur at lower temperatures (Fauske 1992).

NOTE: 4.18 J = 1 cal.

Figure 2-4. Ferrocyanide Tank 3-Component Diagram.





3.0 CORE SAMPLING

3.1 DESCRIPTION OF SAMPLING EVENT

Tank 241-C-109 was push-mode core sampled through three risers from September 2, 1992, to September 7, 1992. One segment was expected from each core sample. Core 47 was obtained from riser #6. Core 48 was obtained from riser #7. Core 49 was obtained from riser #2. The core samples from tank 241-C-109 were obtained using a specially designed core sampling truck (CST). The sampling equipment is mounted on a rotating platform on the CST. Access to the interior of the tank is provided by various tank risers. These risers are pipes of various diameters leading into the tank dome from the ground. The riser configuration for tank 241-C-109 is given in Figure 3-1. A review of the tank farm operating records and a field inspection of the tank risers determine which risers can be used in the sampling operation. A riser is opened and the CST is positioned over the riser. The sampler is lowered into the tank through the drill string and pushed into the waste.

The sampler is constructed of stainless steel and is 48 cm (19 in.) long, with a 2.2-cm (7/8-in.) inside diameter, and has a volume of 187 mL (0.05 gal). Tank Farm Operations has determined that sampling events of one or two segments do not require hydrostatic head balance fluid. Therefore, none was used in this operation, which eliminated any potential problems with sample contamination. When a segment is captured by the sampler, it is sealed within a stainless steel liner, and the liner is placed within a shipping cask. The shipping casks are approximately 122 cm (48 in.) tall, 13 cm (5 in.) in diameter, and have 2.5 cm (1 in.) of lead shielding. This degree of shielding and containment protects workers from excessive radiological exposure and prevents any liquids from the sample (or the sample itself) from being lost.

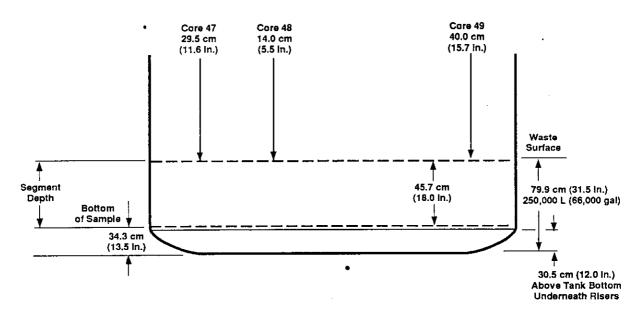
The casks were transported to the 325 Analytical Chemistry Laboratory for characterization analysis. This laboratory is operated by Pacific Northwest Laboratory (PNL) in the 300 Area of the Hanford Site.

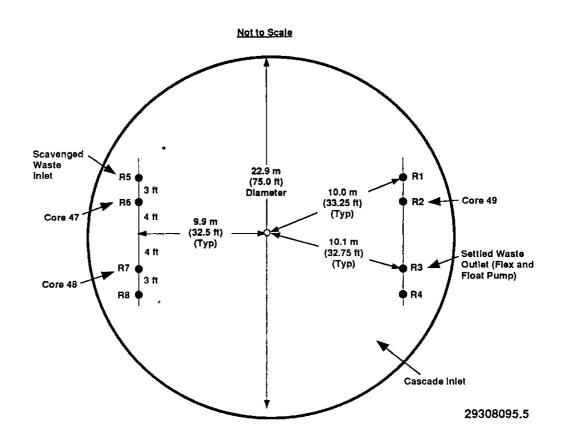
3.2 CHAIN OF CUSTODY

A chain-of-custody record was kept during the sampling event for each segment that was sampled. The chain-of-custody form is a one-page record that is used to ensure that (1) the sample is safely and properly transported from the field to the laboratory, and (2) the correct personnel are involved in the sampling operation and transportation of the sample to the laboratory.

A primary function of the chain-of-custody record is to provide radiation survey data. This is a record of the radiation dose that is emitted from the shipping cask. The dose rates in mrem/hour are measured from the top, sides, and bottom of the cask. These values are recorded on the chain-of-custody form and represent the radiation being emitted directly from the sample. The last item recorded under the radiation survey data is the smearable contamination. Smearable contamination represents the radiation from waste

Figure 3-1. Tank 241-C-109 Riser Configuration.





material that is not sealed within the shipping cask; values greater than 100 mrem/hour are considered unsafe. Measurements are made both in the field and in the laboratory. No smearable contamination was found with these samples.

The chain of custody has several other important functions: (1) to provide a brief description of the cask, sampler, and the expected contents of the sampler (shipment, sample, and cask serial numbers for the specific sampling event); (2) to provide summary information about the analytical suite that the sample will undergo or reference the salient documentation; (3) to provide traceability for the sample during transport; and (4) to ensure sample integrity on arrival at the laboratory. This information is provided to ensure that each sample can be uniquely identified. A summary of the most pertinent data contained in the chain-of-custody forms for the tank 241-C-109 samples is presented in Table 3-1.

Copies of the chain-of-custody forms are on file at the Hanford Analytical Services Management (HASM) office. From inspecting the chain-of-custody records, there appear to be irregularities in the sampling or transport of tank 241-C-109 samples. For example, some liner liquid was found in the Core 47 and Core 49 samples. The liquid found in the liners is assumed to be from the sampler. These irregularities merit a sample integrity concern and potential safety concern (i.e., sample containment was compromised). Further investigation and refinement of the sampler design is in progress.

Table 3-1. Chain-of-Custody Summary.

S1	C 47- 00 000	10 00 070	10 10 00 000	
Sample	Core 47: 92-069	Core 48: 92-070	Core 49: 92-071	
Place taken	241-C-109 Riser 6	241-C-109 Riser 7	241-C-109 Riser 8	
Date taken	9/2/92 to 9/4/92	9/4/92 to 9/6/92	9/6/92 to 9/7/92	
Date released	9/10/92	9/10/92	9/10/92	
Time released	19:20	19:20	19:20	
Sender	L. L. Dean	L. L. Dean	L. L. Dean	
Receiver	T. K. Andrews	T. K. Andrews	T. K. Andrews	
Place received	325 Building	325 Building	325 Building	
Time received	21:45	21:45	21:45	
Smearable contamination	< DL alpha < DL beta-gamma	< DL alpha < DL beta-gamma	< DL alpha < DL beta-gamma	
Dose rate through 1 R/hr the drill string		2.5 R/hr	1.5 R/hr	

<DL = below detection limits</pre>

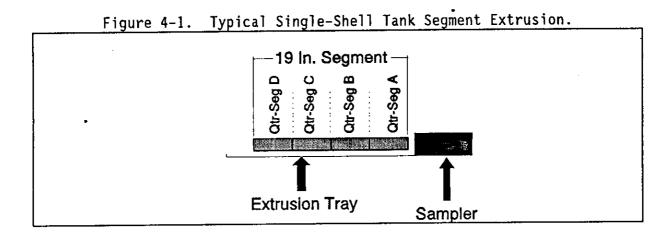
This page intentionally left blank.

4.0 SAMPLE PREPARATION/SAMPLE EXTRUSION

4.1 SAMPLE BREAKDOWN PROCEDURE

Because tank 241-C-109 has been identified as a Watch List tank (as described in Section 2.1.2), more extensive analytical measurements are required to resolve the safety concerns associated with this tank. To enhance the resolution of the assays for key analytes, the analysis horizon for characterization was determined to be one-quarter of a segment.

The sampler was removed from the shipping cask directly into the hot cell. At this time, the sampler is placed into the horizontal position. The sample was then loaded into the mechanical extruder and removed by pushing it out from the back of the sampler with a piston. In this case, the sampler is pressed against a fixed piston, forcing the sample into the extrusion tray. If a full sample has been captured, the material nearest the valve (i.e. the bottom end) was from a deeper part of the tank; the material near the piston was closer to the surface. The sample and any liquids were collected on a metal tray. Next, the mass of the segment and the approximate length were recorded. From this information, the bulk densities of the segments can be estimated. The sample volume is determined by measuring the length of the extruded sample using a linear unit volume of 9.85 mL/in (3.88 mL/cm). Each segment was divided into 12-cm (43-in.) subsegments. Figure 4-1 illustrates how the ferrocyanide SST segment sample was extruded and divided into subsegments. A video record of the extrusions of each of the segments from tank 241-C-109 was made, and color photographs documenting the extruded segments were taken.



Several different styles of nomenclature are used for distinguishing core samples, sample segments, and subsegments in the existing literature. Two major conventions are used in the documentation relating to ferrocyanide (and core sampling in general). The first is designating the segment with the last two digits of the calendar year (92-) and then numbering the segments sequentially (-001, -002, etc.). This system resets itself every calendar year. The second system distinguishes the tank, core, segment, and subsegment. The first (bottom) 12 cm ($4\frac{3}{4}$ in.) of the extruded sample is assigned to the fourth subsegment and is uniquely identified (Tank ID - Core

No. - Segment No. - D). The following three 12-cm ($4\frac{1}{4}$ -in.) sections of the extruded segment are labelled as C, B, and A, respectively. An example of this naming protocol for the third subsegment from the first core is: 241-C-109-C ore 47-S egment 1-B. If the extruded segment is less than 48 cm (19 in.) long, then the same naming convention applies until no solid material is left to make a complete 12-cm ($4\frac{1}{4}-in$.) subsegment. The first 12 cm ($4\frac{1}{4}$ in.) is be assigned to the D subsegment. This second system of naming is the primary convention used in this report. Where no tank identification is given in this report, it should be understood as meaning tank 241-C-109.

4.2 HOMOGENIZATION TESTS

The subsegment and core composite samples are homogenized using a mechanical mixer prior to analysis. This is done so that aliquots removed for analysis will be representative of the entire subsegment or core composite. Aliquots of the homogenized tank waste from Cores 48-1D and 49-1D were taken to determine the efficacy of the homogenization procedure. However, there was not sufficient sample material to perform a homogenization test on Core 47. The samples were split into duplicates, acid digested, and assayed by inductively coupled plasma-atomic emission spectroscopy (ICP) and gamma energy analysis (GEA). This procedure is done to determine if the degree of mixing achieved by the as-planned homogenization procedure was sufficient for the remaining samples to be homogenized and prepared for analysis. If the analytes from the aliquots are within a relative percent difference (RPD) of 10 percent, the samples are considered homogenized. If there are several analytes that are not within the specified RPD, the samples are mixed further and re-assayed. Once homogenization was indicated, the remaining samples were homogenized via the required procedure and prepared for analysis.

Core 48 showed significant differences between the means for the top and bottom segments for several major analytes (Na, Al, Ca, Ni, and P). In addition, large RPDs for between the segment samples were observed for Fe, P. Si, and Mn. Results from the subsegment homogenization test compare well with the core composite values. Core 49 showed no significant differences between the means for the top and bottom segments, but the test did show large RPDs for many elements, with much of the variation occurring in the top sample (RPDs ranging from 21 to 54%). These results indicate that acid digestion as a sample preparation was not appropriate, and that potassium hydroxide (KOH) fusion was required to dissolve this material. This behavior was not unexpected because the simulant materials were very resistant to dissolution. There were adequate amounts of Core 49 material remaining to perform another homogenization test using a fusion dissolution sample preparation. The results from this test indicate that some difference remained between the top and bottom samples for Al and U, with Fe borderline. The RPDs between replicates for each sample were within established acceptance criteria, except for Mn, which is a trace analyte in this sample matrix. The KOH fusion preparation step appears to improve the homogenization test analytes, but the remaining differences between the top and bottom sample means indicate some non-homogeneity in the samples.

4.3 SUBSEGMENT-LEVEL ANALYSES

The objectives of subsegment-level analyses are to provide (1) information as a function of depth pertaining to the overall waste energetics, (2) the distribution of ¹³⁷Cs and ⁹⁰Sr, (3) the concentration and solubility of the CN present in the sample, and (4) a higher resolution for determining bulk tank composition for certain analytes. To accomplish these goals, the limited suite of analyses listed in Table 4-1 was performed on each homogenized subsegment. These analyses were conducted using the analytical procedures identified in Tables I5-1 and I5-2 of WHC-EP-0210, Rev 3 (Hill et al. 1991), and as amended in Hill (1991). Brief descriptions of the sample preparation and assay methods are presented. Laboratory procedures used are described in detail in Simiele (1991).

Table 4-1. Subsegment-Level Analysis.

Direct	Fusion dissolution	Water leach
TOC/TIC TGA DSC Total CN ⁻ Wt% H ₂ O	ICP (Metals) GEA (¹³⁷ Cs) ⁹⁰ Sr	IC (Anions) CN pH GEA

DSC = Differential scanning calorimetry.

GEA = Gamma energy analysis.

ICP = Inductively coupled plasma - atomic emission
 spectroscopy.

TGA = Thermogravimetric analysis.

TIC = Total inorganic carbon.

TOC = Total organic carbon.

Direct analyses are assays performed on the sample matrix with little or no sample preparation. Several direct analyses were performed relating to the energetic properties of the waste; total organic carbon (TOC), scanning thermogravimetric analysis (TGA), DSC, total cyanide, and gravimetric weight percent water.

The TOC was determined using hot persulfate. A sample is dissolved in a sulfuric acid solution (90 °C+) to liberate inorganic carbon (carbonate). Potassium persulfate $(K_2S_2O_8)$ is then added, and organic carbon is converted to CO_2 , which is measured coulometrically. The total organic and total inorganic carbon assays are not considered capable of reliably detecting carbon contained in cyanide compounds in these waste matrices.

Scanning TGA and DSC are useful in determining the thermal stability or reactivity of a material. TGA measures the mass of a sample while the temperature of the sample is increased at a constant rate. In DSC analysis, the heat absorbed/evolved over and above the usual heat capacity of the substance is measured while the substance is exposed to a linear increase in temperature.

Total cyanide analysis was done using a procedure developed at PNL for these specific types of samples. The sample was dissolved in a solution of ethylenediaminetetraacetic acid and ethylenediamine and placed in a microdistillation apparatus. The total cyanide content was determined by argentometric titration.

The gravimetric weight percent water was determined by drying the sample for 12 to 24 hours in an oven at 103 to 105 °C and measuring the difference in the weight of the sample.

Analyses that were performed on fusion-prepared samples were ICP and GEA for radionuclides. Fusion dissolution analyses are assays performed on the sample matrix after it has been fused with potassium hydroxide in a crucible (nicke) crucibles were used) and dissolved in acid. This preparation dissolves the entire sample, whereas other sample preparation procedures may not completely dissolve the sample matrix. However, one significant disadvantage of fusion preparation is that large amounts of potassium hydroxide are required to bring a sample into solution. Because of the high dilution factor, trace elements are less likely to be correctly quantified if they are detected at all. Elements that occur in abundance (major metals) or are highly insoluble are likely to be detected better by the fusion results than by any other sample preparation. Generally, fusion dissolution is the preferred method of analyzing radionuclide content, with the exception of and $^3\mathrm{H}$ (tritium). However, the sample preparation specified in the test instructions for $^{14}\mathrm{C}$ (water digestion) is likely not the best for the ferrocyanide waste. Difficulty with dissolving the sample with a water leach and volatility associated with a fusion preparation will bias the ¹⁴C results low for both sample preparations. An adequate sample preparation method for ¹⁴C is not available for this sample matrix; however, ¹⁴C is not expected to be a significant contributor to the radionuclide content of the waste. *C is not expected to be

Water leach (or water digestion) analyses are assays performed after the sample matrix has been digested in distilled/deionized water; the water is then analyzed for soluble analytes. The soluble anions are determined by ion chromatography (IC). The primary anions analyzed in this manner are fluoride, chloride, nitrate, nitrite, phosphate, and sulfate. In addition, free cyanide and pH were also analyzed from water digestion samples.

4.3.1 Rheological and Physical Measurements

Only one 25-mL aliquot (from Core 47) was used for the full suite of rheological and physical measurements. Viscosity, settling properties, fluid behavior, and shear strength were some of the primary characteristics investigated. The sample tested for these properties was not homogenized prior to analysis. Some selected physical measurements were performed on all of the core composites.

4.3.2 Subsegment Level Archive

Several analyses (adiabatic calorimetry, ferrocyanide speciation, and total oxygen demand [TOD]) have been identified by the Waste Tank Safety Programs as requiring developmental work. A sufficient amount of sample from

each subsegment has been archived to perform these analyses when the procedures for these analyses have been developed. The adiabatic calorimetry assay will be performed on each subsegment if an exotherm of predetermined parameters is detected by DSC analysis. The boundaries for performing adiabatic calorimetry have been determined to be when the DSC exotherm is greater than -75 cal/g and the sample has 15 wt% water or less; or when the exotherm is greater than -125 cal/g, even if the sample has greater than 15 wt% water. Because of sample consumption constraints, the TOD test cannot be run on samples from a subsegment drawn from the same core as a rheology-assayed core.

4.3.3 Core Composite Level Analysis

One composite from each core was built and analyzed in accordance with the complete baseline case core composite scenario detailed in Section 6.1 of WHC-EP-0210 (Hill et al. 1991) and as amended by Hill (1991). The type and number of analytical tests performed are similar to the suite done on the subsegments but are much more extensive. The free liquids from Cores 47 and 49 were combined and analyzed as a separate liquid core composite.

Selected radionuclides were measured on some of the water digestion samples to determine the type and number of water soluble radionuclides. ICP and atomic absorption (AA) spectroscopy were also performed on some of the water digestion samples. These assays were performed to determine the amount of soluble metal cations (ICP) or arsenic, mercury, or selenium (AA). In most cases, these analytes were below the detection limits in the water digestion samples, suggesting that most of the analytes are not water soluble.

Acid digestion is a preparation method where the sample is dissolved in a mixture of nitric and hydrochloric acids. This preparation brings most of the insoluble metals into a solution with a minimum amount of dilution, and is usually best for detecting trace and some major metals. These properties are the reason that acid digestion is generally used as the sample preparation for the homogenization tests. The analyses performed on this preparation were the ICP, GEA, and AA analysis (the AA analysis used nitric acid only). IC analysis was not performed with the acid digestion preparation solution.

Major metals that were well quantified with fusion ICP analysis for tank 241-C-109 were aluminum, calcium, iron, lead, sodium, and uranium. Phosphorous and silicon are non-metallic analytes that were detected by the ICP. In the case of these elements the value from the fusion sample preparation is the more accepted quantity. Although the assay was performed in a nickel crucible, nickel values from the fusion preparation will be reported because they are important to interpreting the overall results. This is done with the understanding that they are biased high. A zirconium crucible was initially recommended for use with these assays to eliminate any potential nickel bias, but the sample matrix reacted with the zirconium during the fusion procedure. Potassium readings from the ICP fusion are not reported because potassium hydroxide was used to dissolve the sample and the potassium results are not important to characterizing the waste. Some of the primary radionuclides that are measured using this sample preparation are neptunium, plutonium, strontium, cesium, and technetium. A total alpha and total beta count were performed on the fusion dissolution samples as well.

A U.S. Environmental Protection Agency (EPA) Contract Laboratory Procedure (CLP) type organics speciation analysis was performed on the core composites. No CLP target compounds or tentatively identified compounds were detected in levels above accepted quantitation limits (HASM 1993) and they were not expected to contribute to the sample matrix.

In previous characterization sampling, the core composites were built using quantities of segments based on a proportion of the total weight of sample for the core (Winters et al. 1990a, 1990b). This method assumed that the sample obtained is representative of what is in the tank. However, when partially filled segments are obtained, this procedure assumes that the tank does not contain any waste in this area. Incomplete recovery for a segment is more likely the result of sampling problems rather than voids in the waste.

The approach used in this analysis effort was to composite equal quantities of the homogenized subsegment material and assume that whatever is obtained in a partial subsegment is representative of a whole subsegment. Some inaccuracies may be introduced from this method because of density differences between subsegments. However, the inaccuracies introduced from density differences would probably be small; those deviations are minimal compared to the other errors inherent in core sampling and analysis. If full segments are obtained for the entire core, and the homogenization procedure is satisfactory, there will be little difference between the two approaches.

5.0 ANALYTICAL RESULTS: TANK 241-C-109

5.1 TANK 241-C-109 CORE SAMPLE RECOVERY

As shown in Figure 5-1, the last 3.8 cm (1.5 in.) of the 48.3-cm (19-in.) sampler does not secure a sample from the bottom segment. In addition, the location of the risers, the dished bottom of the tank, and safety margins in the sampling protocol preclude obtaining samples from the entire waste depth in the tank. Thus, the maximum recovery for the segment from tank 241-C-109 is 3.8 cm (1.5 in.) above the bit bottom to the waste surface. Segment recoveries were based on the maximum recoverable volume for the segment regardless of solid/liquid ratio. The maximum recoverable amount of sample is 45.6 cm (18.0 in.) (177.3 mL). Tables 5-1 and 5-2 present the initial measurements and observations regarding the core samples on extrusion, and an estimate of the core recovery on a volume basis.

Table 5-1. Tank 241-C-109 Core Sample Description Summary.

Core No.	Segment	Core recovery (volume basis)	Total mass (g)	Comments
Core 47	92-069	64.5%	134	Liquid volume was 11 mL; it contained suspended solids. Solids portion was 26.7 cm (10.5 in.) long.
Core 48	92-070	30.6%	73	No liquid captured. Solids portion was 14.0 cm (5.5 in.) long.
Core 49	92-071	87.4%	182	Liquid volume was 22 mL. Solids were medium brown color; Solid segment was 41.9 cm (13.5 in.) long.

NOTE: Sampler linear volume is 9.85 mL/in.

Table 5-2. Tank 241-C-109 Core Sample Physical Characteristics Summary.

Core No.	Segment	Solids sample mass (g)	Liquid sample mass (g)	Solids sample volume (mL)	Liquid sample volume (mL)	Solids bulk density (g/mL)	Liquids bulk density (g/mL)
Core 47	92-069	121	13	103	11	1.2	1.2
Core 48	92-070	73	0	54	0	1.3	N/A
Соге 49	92-071	158	24	128	22	1.2	1.1

Solids = wet solids

Liquid = drainable (free) liquid

NOTE: All liquids were captured in the sample liner.

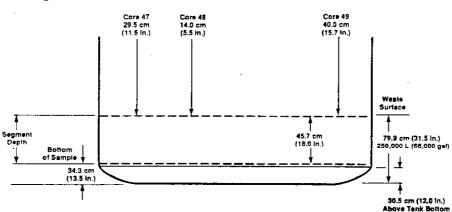
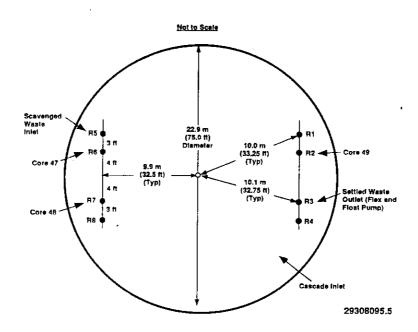


Figure 5-1. Current Waste Level of Tank 241-C-109.



General characteristics of tank 241-C-109 waste materials are as follows.

- Drainable liquids found in the liner were brownish-yellow in color and contained suspended solids.
- Core samples were generally dark brown in color. The brown solids were streaked through with grey/white material.
- The samples had a firm consistency. They were thick, cohesive sludges that held their shape after extrusion. The core materials all appeared to be saturated with liquid.

Analysis of the samples was performed at the PNL Analytical Chemistry Laboratory. The full data package is available from the Hanford Analytical Services Management Office (HASM 1993).

5.2 TWRS PROGRAM ELEMENT CHARACTERIZATION SYNOPSIS

This section provides selected results obtained from core sampling for some of the most pertinent analytes for the various TWRS program elements, including HWVP, Retrieval, Pretreatment, and Waste Tank Safety. Analytes of interest will be reported on a level of resolution commensurate with the available data and program direction. These selections were made on the basis of previous consultation with the program in support of other activities or currently documented characterization data needs (Bell 1993). Watch List tanks will have segment or subsegment level analyses reported, while non-Watch List tanks are analyzed only on a core composite basis. Generally, analytes of interest to multiple programs will only be reported in one section. Further detail can be found in the body of the report or in the data packages.

5.2.1 Retrieval Program Characterization Data Summary: Physical Properties

A major objective of the Characterization program is to measure the physical properties of the waste to support waste retrieval technology development. The physical characteristics of tank waste are required to develop design criteria for waste retrieval equipment, provide a basis for simulated waste development, and to provide a basis for validation of equipment testing using design criteria and simulated waste. The analytical methods to determine the physical properties of the waste as it actually exists in the tank require a substantial amount (50 to 100 g) of unhomogenized sample. In some cases, the limited amount of sample recovered constrains the number of analyses that can be performed.

Performing the rheological/physical measurements once for each stratum of waste in a tank is believed to be sufficient to characterize the entire tank contents. Selected rheological and physical properties are presented in Table 5-3; further information regarding these analytes can be found in Section 5-3.

rable of the revaluation and					
Analyte	Data range				
Specific gravity (g/mL)solidsliquids	1.2 - 1.3 1.1 - 1.2				
Shear strength	17,300 dynes/cm ²				
Viscosity (cP @ 29 °C)1:1 dilution3:1 dilution	80 (high shear) - 4,200 (low shear) 3 (high shear) - 95 (low shear)				
Particle size (μm) Number distribution Volume distribution	89% < 2 μm 70% < 85 μm				

Table 5-3. Retrieval Program.

5.2.2 Pretreatment Program Characterization Data Summary: Bulk Constituent Inventories

The majority of the programmatic decisions pertaining to the design of pretreatment and final disposal systems will be based upon the average characteristics of the tank waste. Therefore, the majority of the laboratory analyses will be conducted on representative core composites. Liquid composites and strata composites will be built under some circumstances and will be analyzed with fewer assays and, as noted previously, segment (or subsegment) level analyses will be performed, when directed.

Table 5-4 presents selected <u>trace</u> analytes of known interest; the Pretreatment Program's data needs are quite extensive and further chemical and radiological characterization information can be found in Sections 5.3, 5.4, and 5.5. Currently, methods are being developed for certain analytes. These developmental assays are then phased in as part of a technology transfer effort. In these cases, samples will be archived until the requisite method has been developed and implemented, or samples will be shipped between the onsite laboratories (222-S Laboratory and 325 Laboratory) and possibly to offsite laboratories for analysis.

Table 5-4.	Trace	Analytes	of	Interest	to	Pretreatment.
------------	-------	----------	----	----------	----	---------------

Analyte	Core composite values			
Minor fusion prep ICP analytes (µg/g) B Cr Mg Zr	Core 47 < DL 270 650 < DL	Core 48 < DL 260 670 < DL	Core 49 < DL 220 340 < DL	
pH (Liquid composite)			12.08	
pH (Water leach)	10.8	10.1	9.4	

ICP = Inductively coupled plasma - atomic emission
spectroscopy.

< DL = below detection limits.

5.2.3 Waste Tank Safety Program Characterization Data Summary

5.2.3.1 Criticality Safety. The criticality safety program has indicated that plutonium and uranium isotopic analyses on each core composite and the bottom most six inches of each core is required to alleviate the concern for the potential of tank criticality (refer to Tables 5-5, 5-6, and 5-7). For the ferrocyanide tanks, the analyses are performed on the bottom subsegment of the core sample. As requested, the analyses will indicate whether the fissile species have settled in a concentrated layer at the bottom of a tank. Therefore, upon extruding the last segment in a core, a small aliquot is taken and analyzed for plutonium and uranium isotopic analyses by mass spectroscopy.

Table 5-5.	Core Composite	Transuranics	(Fusion	Preparation).

Core No.	²³⁷ Np (μCi/g)	²³⁸ Pu* (μCi/g)	²³⁹ Pu* (μCi/g)	²⁴¹ Am _{GEA} (μCi/g)	²⁴¹ Am _{AEA} (μCi/g)	Total α (μCi/g)
Core 47	3.65E-04	4.40E-05	0.82	< 0.58	0.32	0.992
Core 48	3.34E-04	7.15E-06	0.063	< 0.71	0.01	0.065
Core 49	3.01E-04	1.11E-05	0.075	< 0.35	0.13	0.129

 $[\]star$ = Isotopic quantitation of plutonium is determined by multiplying the total Pu α measurement by its mass fraction.

Table 5-6. Core Composite Uranium.

Core No.	U _{ICP} fusion (μg/g)	U _{FL} (μg/g)	²³⁸ U Mass fraction	²³⁵ U Mass fraction
Liquid composite	< DL	3.7	NM	NM
Core 47	9,200	12,000	0.993263	0.006573
Core 48	24,700	27,600	0.993038	0.006852
Core 49	4,700	7,500	0.993109	0.006753

DL = Below detection limits.

Table 5-7. Plutonium Concentration and Isotopic Distribution.

Core No.	Total Pu α (μCi/g)	²³⁸ Pu Mass fraction		²⁴⁰ Pu Mass fraction		²⁴² Pu Mass fraction
Core 47	0.88	0.00005	0.932237	0.066256	0.001216	0.000241
Core 48	0.065	0.00011	0.976356	0.022995	0.000364	0.000176
Core 49	0.079	0.00014	0.949629	0.048786	0.001119	0.000329

NOTE: There was no Pu measurement on liquid composite because the concentration was too low.

AEA = Measurement by alpha energy analysis.

GEA = Measurement by gamma energy analysis.

FL = Uranium measurement by laser fluorimetry.

 $^{{\}sf NM}={\sf No}$ isotopic measurement on liquid composite, concentration too low.

5.2.3.2 Ferrocyanide Tanks. During the 1950's, ferrocyanide compounds were used to scavenge ¹³⁷Cs from the supernate of Hanford Site waste tanks. The potential for an exothermic reaction in the sodium-cesium-nickel ferrocyanide/sodium nitrate complex must be evaluated in waste tanks believed to contain 1,000 gram-moles or more of ferrocyanide precipitates. The characterization objectives in support of resolution of this USQ and in support of retrieval, pretreatment, and final disposal systems design are as follows.

- Determine the waste energetics behavior in the tanks.
- Determine the spatial distribution of fuel, ¹³⁷Cs, and ⁹⁰Sr.
- Determine the concentration of total CN and the speciation of ferrocyanide present in the waste.
- Satisfy the general characterization requirements for physical, chemical, and radiological analytes.

To achieve the above objectives and to enhance the resolution of the vertical distribution study for key analytes, the analysis horizon for characterization of layering is one quarter of a segment. The data from tank 241-C-109 indicates that the tank meets the present criterion for placement on the Ferrocyanide Watch List (i.e., greater than 1,000 g-mole ferrocyanide [estimated from total cyanide measurements]). Further characterization information regarding this topic can be found in Section 5. The next several figures (Figures 5-2, 5-3, and 5-4) present the values most relevant to evaluate the ferrocyanide USQ and provide values for analytes of importance to the Ferrocyanide Safety Program as a function of position.

5.2.4 HWVP Program Characterization Data Summary

The Hanford Waste Vitrification Program has characterization needs in addition to those described for core sampling. Transforming waste into glass is primarily for the disposal of high-level/TRU solids in a geologic repository. The vitrification process will be performed after the solids have been pretreated. Therefore, core sample information will provide preliminary bounding design conditions for the glass plant. Further characterization for technology development and regulatory compliance will be necessary on the pretreated waste that will be fed to the vitrification plant. The analytical requirements for the HWVP program are identified in the Hanford Waste Vitrification Plant Feed Characterization Requirements (Wagner 1992).

Neutralized current acid waste (NCAW) is expected to be pretreated by a sludge washing process prior to becoming the first feed to HWVP. The high heat waste in tanks C-105 and C-106 will also be an early feed to HWVP and is expected to be pretreated in the same manner (one of the two cores from tank C-106 will be analyzed as an early feed tank). Other tanks, such as 241-C-109, will be considered for early retrieval and processing, based on technical and programmatic criteria. Some of the characterization objectives in support of design of retrieval, pretreatment, and final disposal systems are as follows.

Figure 5-2. Core 47 Measurements and Observations.

		alytes			A	nalytes			_
137 Cs μCi g	90 μCi g	239/240 Pu μCi g		H ₂ O wt%	Total CN ⁻ wt%	NO ₂ - wt%	NO ₃ - wt%	ΔH cal/ dry g	– 48 cm
			A						– 36 cm
340	4600	N.M.	В	19.3	0.30	2.8	2.8	No Exotherm	24 cm
770	470	N.M.	С	28.4	0.44	3.7	3.6	No Exotherm	− 12 cm
950	200	N.M.	D	39.4	0.58	4.0	3.9	No Exotherm	– 0 cm
870	1180	0.88	Core Composite	21.5	0.55	3.9	3.7	No Exotherm	

Drainable Liquid N.M. = No Measurement

29308095.2

Solids

Figure 5-3. Core 48 Measurements and Observations.

Analytes			A	nalytes			_
$ \begin{array}{c c} 137 & 90 & 239/240 \\ Cs & Sr \\ \underline{\mu Ci} & \underline{\mu Ci} & \underline{\mu Ci} \\ \hline g & g & g \\ \end{array} $	Pu	H ₂ O wt%	Total CN ⁻ wt%	NO ₂ - wt%	NO ₃ - wt%	ΔH cal/ dry g	– 48 cm
	A						– 36 cm
	В						– 24 cm
1200 150 N.M.	C	53.0	1.13	5.1	5.6	N.M.	− 12 cm
1170 120 N.M. 1030 190 0.065	Core Composite N.M. = No Measureme	52.0 57.7	0.87	5.0 4.5	5.4 4.8	-12.4 N.M.	- 0 cm

Solids

Figure 5-4. Core 49 Measurements and Observations.

	Analytes				Analytes				
137 Cs μCi g	90 Sr <u>μCi</u> g	239/240 Pu <u>µCi</u> g		H ₂ O wt%	Total CN* wt%	NO ₂ - wt%	NO ₃ - wt%	ΔH cal/ dry g	- 48 cm
			A						– 36 cm
120	2400	N.M.	В	19.6	0.35	2.7	2.6	N.M.	– 24 cm
350	200	N.M.	C	38.3	0.81	4.4	4.2	N.M.	- 12 cm
700 560	190 930	N.M. 0.079	D Core Composite	39.6 27.8	0.55 0.56	4.5 3.9	4.3 3.6	N.M. N.M.	- 0 cm
	nable Lie				}		00	20005 4	Į

Drainable Liquid N.M. = No Measurement

29308095.4

Solids

- Provide extensive characterization of the chemical and radiological contents of the waste (solids and supernate) as it currently exists in the tanks to support processibility assessments and to verify whether the composition variability study envelope coverage for key analytes is adequate.
- Provide sufficient data to make an estimate of the waste fraction that will remain after sludge wash pretreatment and become feed for the HWVP.
- Determine the physical and rheological properties of the waste before and after simulated sludge washing to support the design of a waste retrieval system.
- Satisfy the general characterization requirements for physical, chemical, and radiological analytes.

Planned early retrieval of some tanks for HWVP necessitates an increased evaluation of rheological and physical properties of the tank waste. To this end, selected rheological and physical properties are measured on the first and last segments of both cores taken from these specified tanks as a minimum. Rheological measurements will also be performed on other segment material if a unique stratum is identified in the remaining segments.

The analytical program for HWVP not only entails determining whether a waste type is suitable for disposal as glass, but also includes determining the physical and chemical characteristics of the glass for process control purposes and to ensure regulatory compliance. Sampling and analysis plans will be developed on an individual basis for each tank or process batch. The characterization needs for these efforts include analyses for metals, water-soluble anions, radionuclides, semi-volatile organics, and rheological and physical testing for both the HWVP feed and vitrified product.

Presently, tank 241-C-109 is not scheduled as an early feed to the HWVP. However, in recognition that the tank may be considered in the future as a potential early retrieval processing candidate, two selected groups of analytes are presented in this summary; Table 5-8 provides a set of analytes of interest to the vitrification process control, the other (Table 5-9) are analytes of interest to the regulatory permitting of such a facility.

Analyte	Core 47 composite (µg/g)	Core 48 composite $(\mu g/g)$	Core 49 composite (µg/g)
PO ₄ 3- (IC/ICP)	22,100/61,700*	26,700/62,600*	12,800/45,300*
F.	400	1,300	400
C1 ⁻	700	800	700
TOC	2,300	3,100	2,300

Table 5-8. HWVP Process Control Analytes of Concern.

^{* =} based on an ICP fusion value for phosphorous and assuming it is present solely as phosphate.

Analyte (μg/g)	Core 47	Core 48	Core 49
Hg (Liquid composite)	8.9	6.6	6.7
Pb (Liquid composite) < DL (Solid composite, fusion)	N.M. 7,300	N.M. 700	N.M. 820
Cr(VI) (Solid composite, water)	47	37	28*
Analyte (μCi/g)	Core 47	Core 48	Core 49
14C (Liquid composite: 2.3E-03) (Solid composite, water)	6.3E-06	1.8E-05	3.6E-05
99TC (Liquid composite: 1.6E-01) (Solid composite, fusion)	1.1E-01	1.2E-01	9.4E-02
³ H Liquid composite: 3.29E-03) (Solid composite, water)	8.5E-03	6.4E-03	6.4E-03

Table 5-9. HWVP Regulatory Operation Analytes of Concern.

5.3 TANK 241-C-109 CORE SAMPLE RHEOLOGICAL/PHYSICAL MEASUREMENTS

Physical properties such as shear strength, viscosity, particle size, and settling properties were measured. These measurements are necessary for the design and fabrication of retrieval, pretreatment, and final waste disposal systems.

5.3.1 Shear Strength

The shear strength of the waste from tank 241-C-109 was measured on a combined, unhomogenized sample from Core 47. The shear strength measurements were made at ambient temperature using a shear vane connected to a viscometer and rotated at 0.3 r/min. Shear strength $(\tau_{\rm s})$ is a semiquantitative measurement of the force required to move the sample. Because shear strength is dependent on sample handling, the measurement was taken without any sample homogenization. The rheology sample was generated by taking small aliquots from the segment of Core 47 at various positions. The aliquots were transferred to a sample jar and allowed to settle for 10 weeks to let the sample recover from the disturbance of sampling and extrusion. The extended delay between sample and analysis was specified because it is believed that the longer the sample sits undisturbed, the more likely it is to return to its (nearly) original condition; therefore, the shear measurement is likely to be more representative. The shear stress $({\rm S_{\tau}})$ of the sample was recorded as a function of time and the shear strength was calculated using Equation 1.

^{*}Single data point; other anomalous data point is out of range.

$$\tau_{s} = \frac{\left[8\tau/100 \right] * S_{\tau} * 4.9E + 05}{\frac{\pi * H_{v} * D_{v}^{2}}{2} + \frac{\pi * D_{v}^{3}}{6}}$$
(1)

where:

 $\%\tau/100$ = The ratio of the total torque which is recorded as full scale on the plot of the shear stress

 $S_{\tau} = Shear stress$

4.9E+05 = maximum torque of the viscometer head (dynes)

 $H_v = \text{Shear vane height } (0.800 \text{ cm } [0.315 \text{ in.}])$

 D_{ν}^{ν} = Shear vane diameter (0.803 cm [0.316 in.]).

Two measurements of τ_s were taken, averaging 17,300 dynes/cm² (17,560 and 17,000 dynes/cm²). The shear stress of the material exceeded the maximum value for the measurement system (8,500 Pa). However, to take a measurement, the core was rotated at a significantly higher rate than was used in the τ_s measurement, causing the measured shear stress to be higher than the actual value. In addition, some drying of the sample may have occurred, also causing the measurement to be higher than its true value.

5.3.2 Shear Stress and Viscosity as Functions of Shear Rate

Shear stress and viscosity measurements (as functions of shear rate) were performed on the 1:1 (water:sample) dilution of the sample at ambient hot cell temperatures 29 to 32 °C (84 to 90 °F) and at 95 °C (203 °F). Drying of the sample at 95 °C posed difficulties in measurement for the 1:1 diluted sample; no results of the rheograms run under these conditions are presented.

The data from the rheograms for the 1:1 dilution were fit to a nonlinear yield power-law model (Equation 2). Sample and duplicate measurements were run at ambient and 95 $^{\circ}\text{C}$.

$$S_{\tau} = \alpha + \beta \gamma^{n}$$
 (2)

where:

 $S_{\tau} = \text{shear stress}$

 α = yield stress (not a fit parameter)

B = consistency factor

 $y = \text{shear rate } (0 \text{ to } 468 \text{ s}^{-1})$

n = flow behavior index.

Table 5-10 presents the power law model parameters for the 1:1 sample dilutions at 30° C.

Sample	Temperature (°C)	Trial	α, Yield stress (Pa)	ß, Consistency factor (Pa•s)	n, Flow behavior index
1:1 Dilution	30	S	50	0.017	1
1:1 Dilution	30	D	40	0.019	1

Table 5-10. Power-Law Model Parameters for Tank 241-C-109 Material.

Viscosity of the 1:1 diluted sample at low shear (i.e., near zero) ranged between 2,800 and 4,200 cP; the viscosity gradually declined with increasing shear rates to 100 cP @ 468 s⁻¹. The 1:1 dilution of the composite sample exhibited yield-pseudoplastic behavior. Figures 5-5 and 5-6 present data smoothed results for shear stress versus shear rate and viscosity versus shear rate for the 1:1 diluted sample. The 3:1 dilution samples exhibit near Newtonian behavior at the detection limit of the system (2 cP) for shear stress as a function of shear rate. Viscosity of the 3:1 diluted sample at low shear ranged between 12 and 42 cP (avg. ~30 cP); the viscosity rapidly declined with increasing shear rates to approximately 5 cP @ 100 s⁻¹ and 3 cP @ 468 s⁻¹. Higher viscosities at higher temperatures for these sample matrices is not unusual, because drying of the sample often has a significant impact on its flow behavior.

Further measurements of the viscosity as a function of shear rate were made on the 3:1 dilution samples at 95 °C (203 °F). The 3:1 dilution samples exhibit near Newtonian behavior at the detection limit of the system (2 cP) for shear stress as a function of shear rate. Viscosity of the 3:1 diluted sample at low shear ranged between 37 and 95 cP (avg. \sim 58 cP). The viscosity rapidly declines with increasing shear rates to between 5 and 12 cP @ 100 s and approximately 3 cP @ 468 s \sim 1.

A rheogram for a material with a yield stress has two sections. The first section is a straight line beginning at the origin and climbing up the ordinate. This portion of the rheogram records the material as it acts like a solid or gel. When sufficient force is applied to the material to make the gel yield, the rheogram breaks sharply to the right and records the material's behavior as a fluid. The point on the rheogram at which the sample's behavior transfers from a solid or gel to a fluid is the yield point or yield stress. The consistency factor in this model is analogous to viscosity. The flow behavior index indicates the degree of deviation from Newtonian behavior. For values less than 1, the behavior is considered pseudoplastic (Bird et al. 1960). Plots of all of the measurements can be found in the full validated data packages (HASM 1993).

5.3.3 Slurry Flow Properties

Turbulent flow is necessary to keep particles in suspension and prevent the accumulation of the solids in retrieval and/or pretreatment process

S = Sample

D = Duplicate

Figure 5-5. Shear Stress Versus Shear Rate for 1:1 Diluted Sample.

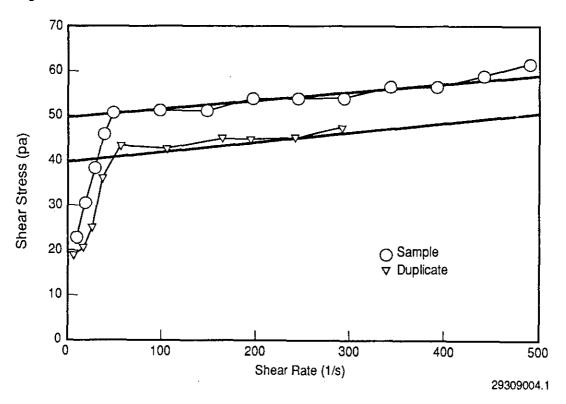
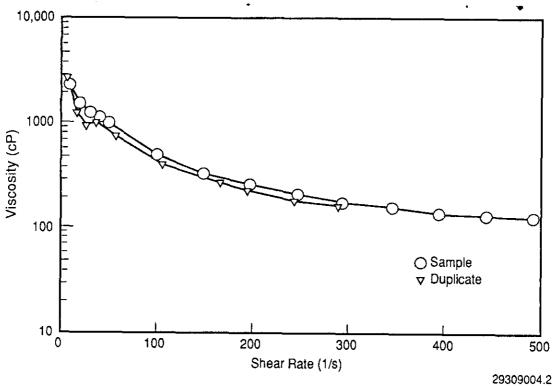


Figure 5-6. Viscosity Versus Shear Rate for 1:1 Diluted Sample.



equipment. Characteristics necessary for turbulent flow were calculated for the 1:1 dilution slurry using the parameters determined from measurement and a curve-fitted rheological model (HASM 1993) (refer to Table 5-11).

Sample	Temperature (°C)	Trial	Pipe diameter (in.)	Velocity (m/s)	Critical flow rate (L/min)	Reynolds number
1:1 Dilution	30 (86 °F)	S	2 (5.08 cm)	3.26	424	12,800
1:1 Dilution	30 (86 °F)	D	2 (5.08 cm)	3.14	405	16,900
1:1 Dilution	30 (86 °F)	S	3 (7.62 cm)	2.90	833	16,900
1:1 Dilution	30 (86 °F)	D	3 (7.62 cm)	2.77	799	14,400

Table 5-11. Turbulent Flow Model Calculations.

5.3.4 Particle Size Measurement

Particle size analysis is performed by placing a small amount of sample in a dispersant, which is the liquid used to disperse and suspend the particles from the solid sample. Samples from all three cores (Cores 47, 48, and 49) were prepared and assayed. The prepared sample was placed in a particle size analyzer. The apparatus measures particle size by passing a thin beam of laser light through the dispersant. The diameter of a particle of matter in the dispersant can be determined by the amount of light that it blocks as the particle passes through the beam. The dimension measured by this method is the value across the short diameter of the particle. This means that if a particle is oblong, the machine estimates the shortest length across the particle (i.e., the width of the oblong shape, not the length). The term "diameter" throughout this text will be used to describe any linear profile of any shape.

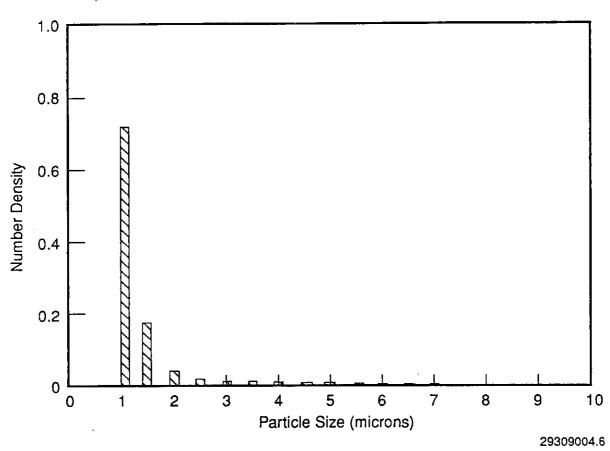
An important consideration involving the analysis of particle size is the dispersant used. The primary concern involved with the dispersant is dissolving the particle. Any particles existing in the tank that are soluble in the dispersant will dissolve or decrease in size during the analysis. Depending on the dispersant, the particle size analysis may not represent the true particle size distribution in the tank. In the case of tank 241-C-109, a mixture of water and glycerin was used as the dispersing medium. If a "true" particle size distribution is required, the mother liquor (drainable liquid) of the tank should be used, if possible, because the tank particulates are already in equilibrium with the tank mother liquor. The insolubility of the waste matrix suggests that the particle size data acquired should be acceptable. However, if the ferrocyanide waste has been hydrolyzed by high-pH waste, this assumption may not be completely accurate.

The mean particle size in the number distribution ranges from 0.80 to 1.38 microns in diameter for tank 241-C-109 waste samples. Table 5-12 presents the summary results of the measurements. Plots of the probability number density for each core are presented in Figures 5-7, 5-9, and 5-11 as a number fraction. The number density graph is plotted over the acquisition

S = Sample

D = Duplicate.

Figure 5-7. Core 47, Particle Size Number Density.



range of the device (from 0.5 to 150 $\mu m)$. The numbers of particles in each size range (shown as a percentage of the whole) are graphed against their respective size ranges to form a distribution curve. It can be seen from the figures that the most common occurrences (modes) for particle size range between 0.5 and 1.0 microns. The majority (over 88 percent) of the measured particles fit within the narrow band of 0.0 to 2.0 microns.

Table 5-12. Particle Size Distribution by Number: $89\% < 2 \mu m$ (all cores).

Sample	Mean (μm)	Median (μm)	
Core 47	1.14	0.85	
Core 48	0.80	0.77	
Core 49	1.38	0.90	

The particle size in the volume distribution ranges from 0.0 μ m to 70 μ m in diameter between the three cores with relatively wide variation between the means of the these samples (5.73 to 37.56 μ m). Table 5-13 presents the summary results of the measurements. Under the assumption that the density of the solid material within the tank is constant, the volume distribution is also the best estimation of the mass particle size distribution of the tank. The analyzer calculates particle volume as the cube of the diameter. These distributions are presented as Figures 5-8, 5-10, and 5-12.

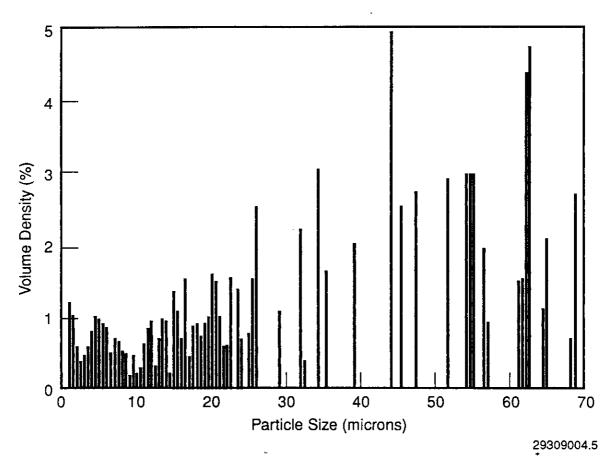
Table 5-13. Particle Size Distribution by Volume: $100\% < 70 \mu m$ (all cores).

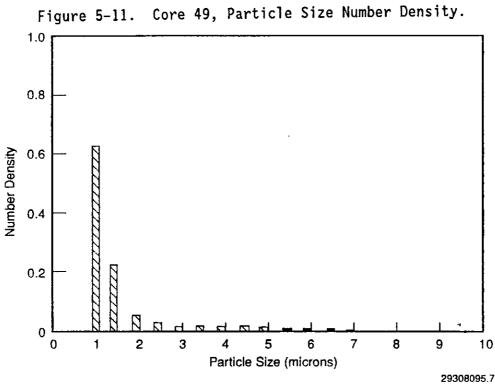
Sample	Mean (μm)	Median (μm)
Core 47	37.56	38.72
Core 48	5.73	2.97
Core 49	24.47	24.08

The volume distribution is represented by a percentage on a probability volume density graph. The average particle size represented in the volume distribution is considerably larger than that in the number distribution. In Core 47 there are relatively few small particles, as most of the particle volume is evenly dispersed within the 0.0 to 70.0 μm range. In Core 48 the majority of the particles are much smaller, with particle volumes concentrated in two narrow ranges, the 0.0 to 2.0 μm range, and the 9.0 to 20.0 μm range. Core 49 is quite similar to core 47, in that there are relatively few small particles, most of the particles are evenly dispersed within the 10.0 to 60.0 μm range.

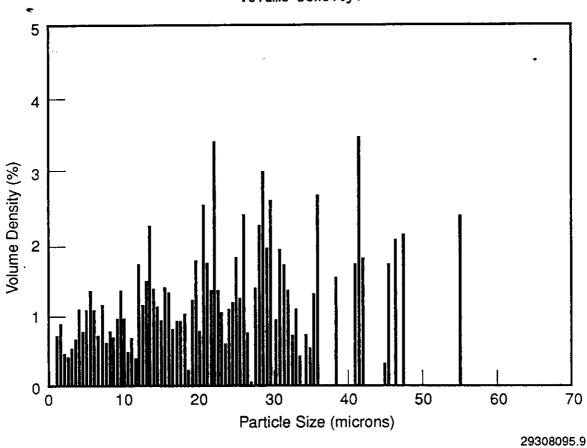
The disparity between the core sample measurements possibly indicates a difference in waste type. In Core 48, over 50 percent of the volume is contained in particles with a diameter of less than 3 μ m. In the Core 47 and 49 samples over 50 percent of the particle volume is contained in material with a diameter of greater than 20 μ m. In the retrieval and subsequent

Figure 5-8. Single-Shell Tank Core 47, Particle Size Volume Density.





Single-Shell Tank Core 49, Particle Size Volume Density. Figure 5-12.



5-20

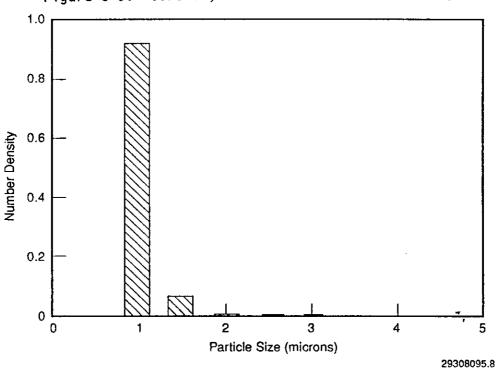
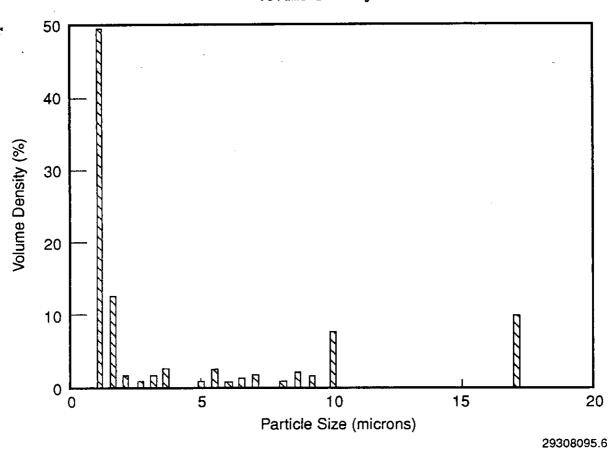


Figure 5-9. Core 48, Particle Size Number Density.

Figure 5-10. Single-Shell Tank Core 48, Particle Size Volume Density.



treatment of the tank wastes, it may be desirable to design pumping or filtration systems for the tank particulate. Therefore, the volume distribution of the particles should not be neglected (i.e., particles with diameters of over 20 $\mu \rm m$ should be considered in these designs). In addition, the behavior of the particle size distribution is believed to have an impact on analytical precision, especially with small sample sizes and thus should be considered when evaluating analytical results.

5.3.5 Settling Behavior of As-Received and Diluted Samples

This section analyzes the settling behavior for the as-received, 1:1, and 3:1 water:sample dilutions. The physical properties reported here include settling rates and volume percent settled solids, and weight percent and volume percent centrifuged solids. The experimental procedures used to perform these measurements were reported previously (HASM 1993).

The physical properties of the Core 47 material and diluted samples are summarized in Table 5-14.

Table 5-14. Physical Properties Summary.

		Segment		
Property	As-Received	1:1 Dilution	3:1 Dilution	
Settled solids (vol%)	100%	88%	41%	
Centrifuged solids		<u> </u>		
Volume %	100%	NM	21.1	
Weight %	100%	NM	27.0	
Density (g/ml)		<u> </u>	`	
Sample* solid liquid	1.2 - 1.3 1.1 - 1.2	NM	1.11	
Centrifuged supernate	NM	NM	1.01	
Centrifuged solid	NM	NM	1.39	

NM = No measurement.

Because there was no free liquid with the waste in the sampler, no settling was observed in the as-received segment samples over a period of three days and there was no standing liquid obtained with the samples. Two dilutions (1:1 and 3:1 water to sample) were prepared, and the volume percent of settled solids for each of the dilutions are plotted as a function of settling time. Figures 5-13 and 5-14 illustrate the setting behavior over time.

The 1:1 dilution reaches a final volume percent settled solids of 88 percent (avg.). Settling continues throughout the 3-day period, but the majority of the settling is seen in the first 24 hours. The 3:1 dilution reaches a final volume percent settled solids of 41 percent (avg.). The majority of the solids settling is complete within 10 hours.

^{*}Obtained from bulk measurements.

Figure 5-13. Settling Rate Data for Tank 241-C-109 Core 49, 1:1 Dilution.

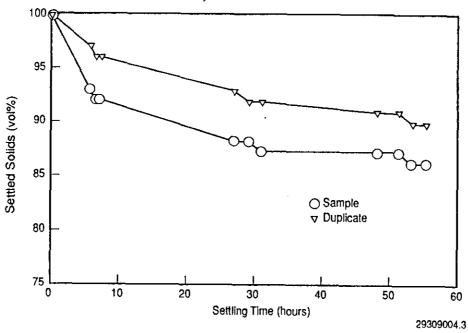
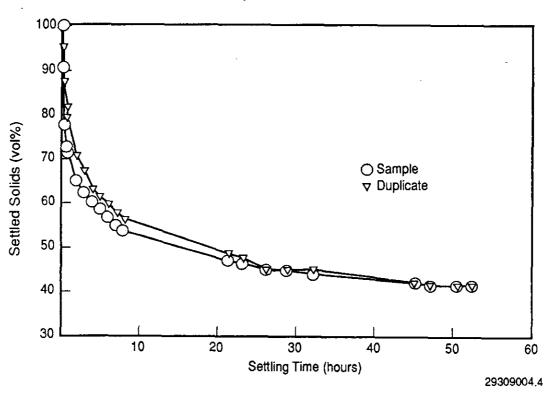


Figure 5-14. Settling Rate Data for Tank 241-C-109 Core 49, 3:1 Dilution.



5.4 ANALYTICAL RESULTS—INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY

5.4.1 Inductively Coupled Plasma Assays—General Comments

No online multiple inter-element corrections were performed for matrix interferences. The ICP has built-in correction capability to adjust for moderate matrix interferences. However, this function suffers from significant performance degradation on samples containing weight percent quantities of iron, aluminum, or uranium. Single pass offline corrections were performed to correct for high aluminum, iron, and uranium content in the samples. Corrections for other interfering analytes were done as required. As requested, process blank values have not been subtracted from the reported values (except for quality control results). In the water digestion and liquid composite assays, the single most prevalent element is sodium, by at least an order of magnitude. In the fusion assays, some elements can appear to be at high concentrations because of the large dilution factors required for fusion samples and the complicating factor of the matrix interferences. Those analytes may actually only be present in concentrations marginally above the detection limit. Calcium, selenium, and silicon routinely demonstrated a low bias (i.e., were present at higher concentrations than reported). While selenium is not a major analyte in the waste matrix, calcium and silicon are. and this behavior could marginally affect the interpretation of the results. An estimate of the detection limit for any analyte can be obtained by multiplying the analyte's DL (detection limit factor, based on dilution) by the appropriate sample " μ g/g factor" found in the data packages. Analytes reported in the data tables are those consistently contributing significant amounts (i.e., generally greater than 0.2 weight %) to the composition of the waste matrix; average values for the analytes are reported to three significant figures based on two replicates. The full range of ICP analytes can be found in the full data packages (HASM 1993). All reported concentration values are based on grams of wet sample, unless otherwise specified.

Tables 5-15, 5-16, and 5-17 provide ICP analyte concentration information on the core composites as a function of the sample preparation.

Table 5-15. Water Digestion Chemical Composite Data -- ICP Average Values.

Analyte	Core 47 (μg/g sample)	Core 48 (µg/g sample)	Core 49 (µg/g sample)	Liquid Composite*
Al	410	110	105	160
Ca	180	60	80	210
Fe	880	1,140	920	1,680
Na	67,800	83,500	59,900	96,900
Ni	130	30	50	340
P	6,990	8,690	4,160	4,200
Pb	< DL	< DL	< DL	< DL
Si	120	200	70	70
U	< DL	< DL	< DL	< DL

*The liquid composite is composed of 11 mL of liner liquid from Core 47 and 22 mL of liner liquid from Core 49.

< DL = below detection limits.

Table 5-16. Acid Digestion Chemical Composite Data -- ICP Average Values.

Analyte	Core 47 (µg/g)	Core 48 (μg/g)	Core 49 (μg/g)
Al	72,900	6,400	83,700
Ca	20,000	12,600	12,300
Fe	28,800	20,200	7,100
Na	81,900	87,600	81,300
Ni	14,700	15,500	11,900
Р	18,400	17,000	19,400
Pb	8,600	610	900
Si	1,900	1,300	1,400
U	10,700	15,000	6,300

Table 5-17. Fusion Digestion Chemical Composite Data --ICP Average Values.

Analyte	Core 47 (µg/g)	Core 48 (µg/g)	Core 49 (µg/g)	
Al	116,800	8,600	126,600	
Ca	24,700	17,700	14,900	
Fe	21,800	22,200	9,100	
Na	87,200	100,000	76,500	
Ni*	31,900	33,000	22,900	
P	19,900	20,200	14,600	
Pb	7,300	700	820	
Si	15,800	2,200	2,300	
U	9,200	24,700	4,700	

Tables 5-18, 5-19, and 5-20 provide ICP analyte concentrations as a function of depth, i.e., for the subsegments. Subsegment samples labeled "B" are at the top of the tank; samples labeled "C" and "D" are progressively deeper.

Table 5-18. Tank 241-C-109 Core 47 ICP Analyte Trending (Fusion Prep on Subsegments).

Subsegment	Al (μg/g)	Ca (μg/g)	Fe (μg/g)	Na (μg/g)	Ni* (μg/g)	P (μg/g)	Pb (μg/g)	Si (#g/g)	(#8/8)
1B	131,600	10,400	63,400	51,100	19,400	7,600	5,100	18,700	11,800
10	120,300	18,000	20,900	63,100	22,700	12,500	2,900	6,100	6,100
1D	32,000	28,000	15,300	102,600	25,600	30,100	14,300	22,200	5,800
Core Comp.	116,800	24,700	21,800	87,200	31,900	19,900	7,300	15,800	9,200

*Nickel concentrations are biased high. Values are derived from ICP fusion performed in a nickel crucible. However, in each case the blank value was an order of magnitude (or greater) less than the measurement. The fusion values are a factor of two greater than the acid digestion values, where both are available.

10

Core Comp.

	(Fusion Prep on Subsegments).										
Subsegment	Al (μg/g)	Ca (μg/g)	Fe (μg/g)	Na (μg/g)	Ni* (#g/g)	P (μg/g)	Рb (#g/g)	Si (µg/g)	υ (μg/g)		
C	7,300	29,300	20,000	115,800	44,200	23,300	550	2,900	16,700		

102,000

100,000

24,000

33,300

20,900

20,200

690

700

2,200

2,200

14,400

24,700

21,000

22,200

Table 5-19. Tank 241-C-109 Core 48 ICP Analyte Trending (Fusion Prep on Subsegments).

Table 5-20. Tank 241-C-109 Core 49 ICP Analyte Trending (Fusion Prep on Subsequents).

Subsegment	Αl (p/gμ)	Ca (µg/g)	Fe (μg/g)	Na (μg/g)	Ni* (μg/g)	P (μg/g)	Pb (μg/g)	\$i (#g/g)	U (μg/g)
18	185,300	4,300	15,600	43,000	10,750	4,100	1,990	2,900	7,900
1C	95,800	18,600	4,600	62,900	31,900	11,500	370	880	1,300
10	70,900	22,500	15,400	91,400	30,700	20,400	730	1,680	12,400
Core Comp	126,900	14,900	9,100	76,500	22,900	14,600	820	2,300	4,700

*Nickel concentrations are biased high. Values are derived from ICP fusion performed in a nickel crucible. However, in each case the blank value was an order of magnitude (or greater) less than the measurement. The fusion values are a factor of two greater than the acid digestion values, where both are available.

5.4.2 Liquid Core Composite

9,800

8,600

16,800

17,700

Comparing the results of the ICP assays between the water leach of the core composites (47, 48, 49) and the liquid composite sample (Table 5-13), the results were found to be somewhat similar. However, because the drainable liquid is composed of liquids assumed to be in equilibrium with material from Cores 47 (11 mL) and 49 (22 mL) assayed with an acid digestion preparation and that the water leach of the core composites is done at a 100:1 dilution, the similarity is only superficial. The full suite of quality control was performed on the liquid composite sample. While the RPDs were acceptable for all major analytes and percent recovery for the control sample was very good (87.9 percent or better); the spike concentration for ferrocyanide, iron, sodium, and nickel was insufficient for percent recovery quantitation.

5.4.3 Core 47

The water leach of the core composite, as noted previously, is similar to the liquid core composite. Major analytes present include sodium and phosphorous, with much lesser amounts of calcium, iron, and nickel. Sodium was approximately one-third less than that measured in the liquid composite, while phosphorous was greater by about one-third. The RPDs were acceptable for all major analytes.

The results from the acid digestion preparation of the core composite samples (Table 5-16) are very good in general for most of the major analytes. Sodium, aluminum, calcium, sodium, nickel, uranium, and phosphorous are all

well within acceptable precision criteria. However, iron, silicon, and lead are all significant contributors to the waste matrix and are outside of the typical 20 percent precision criteria, with the sample showing unexpectedly elevated levels of silicon and lead. Iron particularly stands out, with an RPD of ~60 percent. The lower duplicate value (22,400 ppm) agrees well with the fusion results (20,200 ppm and 23,400 ppm). Because most of the other major elements possess relatively good RPDs, which indicates a reasonably homogeneous sample, the anomalous result was attributed to the presence of a particle inclusion (such as rust) in the sample, thus the high iron result should be considered suspect. The lead and silicon results are much higher in Core 47 than the other cores; the acid and fusion core composite results for lead are comparable. Recoveries of spike control are generally between 80 percent and 120 percent, except for calcium, selenium, and silicon which demonstrate a low bias. However, the sample spike recoveries for several major and minor analytes indicate that the spiking level is inappropriate.

The results from the fusion preparation core composite and subsegment samples are good. The RPDs are within acceptable tolerances for all major analytes. Fusion dissolution appears to be necessary to obtain quantitation of aluminum, calcium, sodium, and silicon. Lead, and silicon remain unusually high in this core as compared with the other two cores (concentrations for these analytes are much higher in this core than Cores 48 and 49). Elevated aluminum and silicon values suggest the presence of alumina and silica, but without any other significant changes in the remaining metals, there is no evidence for a refractory-metal-alumina silicate. Nickel also appears to show a high bias (as anticipated from the assay method, using a nickel crucible). The subsegment assays also demonstrate good RPDs for all major analytes except for iron throughout (and lead in subsegment 1D). Iron routinely demonstrates poor agreement (i.e., RPDs greater than 20 percent) between the sample and the duplicate in this core.

5.4.4 Core 48

As noted previously, the water leach of the core composite is generally similar to the liquid core composite (Table 5-15). Major analytes present include sodium and phosphorous, with much lesser amounts of calcium, iron, and nickel. Levels of sodium and iron are very comparable to the liquid composite results. The RPDs were acceptable for all major analytes, except for phosphorous (74.3 percent).

The acid digestion results for the Core 48 composite correlate very well with the fusion results (Tables 5-16 and 5-17). The percent differences between the 1-fold and 10-fold dilutions in the duplicate are very close, as was found in Core 47. However, agreement between sample and duplicate for several major analytes (iron, phosphorous, uranium, and calcium) is outside the accepted 20 percent precision criteria. Furthermore, this may indicate a significant sample inhomogeneity. This behavior was also encountered in sample material obtained from tank 241-C-112. The analyte distribution for this sample is quite different from Cores 47 and 49. The sodium concentration is much higher than aluminum in this sample and the lead and aluminum levels in Core 47 are higher in general by an order of magnitude.

The sample/duplicate agreement from the fusion preparation core composite and 1C subsegment samples are generally poor (i.e., significantly outside the accepted 20 percent precision criteria). The RPDs are not within acceptable tolerances for several major analytes (aluminum, uranium, and phosphorous in the core composite; iron, sodium, silicon, and phosphorous in the 1C subsegment). The subsegment 1D assay RPDs are acceptable for all major analytes, except iron. While the good correlation of acid to fusion results indicate that there is little refractory material in this core, fusion dissolution appears to be necessary to obtain quantitation of sodium, silicon, and uranium. Aluminum content is much lower and uranium is slightly higher than that found in either Core 47 or Core 49. Nickel is again biased high from corrosion of the crucible during the assay. Lead is low compared to Core 47 but comparable to Core 49. The overall analyte profile appears much more similar to samples taken from tank 241-C-112 than to the other two cores taken from tank 241-C-109.

5.4.5 Core 49

As noted previously, the water leach of the core composite is similar to the liquid core composite. Major analytes present include sodium and phosphorous, with much lesser amounts of calcium, iron, and nickel. Sodium was approximately 40 percent less than that measured in the liquid composite, while phosphorous was about the same. The RPDs were acceptable for all major analytes.

The results from the acid digestion preparation of the core composite samples are consistently poor for all of the major analytes (sodium, aluminum, calcium, sodium, nickel, iron, silicon, lead, uranium, and phosphorous). All of these analytes are far outside of the typical 20 percent precision criteria. The large RPDs seen in this sample indicate inhomogeneity and strongly suggest that this sample preparation/assay is not adequate to provide quantitative results for this sample. However, some general trends can be determined by comparison with other cores. Aluminum is high, comparable to Core 47; iron and uranium concentrations are lower than those in the other two cores; and the lead levels are at the same level as Core 48. The recoveries of the spike control are generally between 80 percent and 120 percent, except for calcium, selenium, and silicon which demonstrate a low bias. However, the sample spike recoveries for several major and minor analytes indicate that the spiking level is inappropriate.

The sample/duplicate agreement from the fusion preparation core composite and subsegment samples are acceptable. The RPDs for the core composites are within established tolerances for all major analytes, except phosphorous. Fusion dissolution appears to be necessary to obtain quantitation of aluminum and silicon. Calcium, lead, and silicon are much lower in this core as compared with Core 47 (concentrations of these analytes are closer to Core 48). Elevated aluminum and silicon values suggest the presence of alumina and silica, but without any other significant changes in the remaining metals, there is no evidence for a refractory-metal-alumina silicate. Nickel also appears to show a high bias (again, as anticipated from the assay method, using a nickel crucible). The subsegment assays also demonstrate good RPDs for all major analytes except for iron and calcium (in subsegment 1B).

5.5 ANALYTICAL RESULTS—ANION ASSAYS

5.5.1 Ion Chromatography Assays---General Comments

IC analyses were performed on water-leached samples of the subsegments, core composites, and diluted samples of the liquid composite. Sample blanks indicate no anion contamination from the 325-A hot cell. Because matrix components in some of the samples were found to affect detector performance reversibly during the analysis for free cyanide, a modification to the procedure using pulsed electrode cleaning between sample injections was incorporated to overcome that effect. In addition, the free cyanide assay produced much higher results than those anticipated from the simulant studies. No spike or control standard was used for the free cyanide analysis, as specified in the procedure (Simiele 1991). Therefore, these results may not be representative of the free cyanide in the samples. Quantitation for fluoride (and possibly chloride) was compromised by a co-eluting matrix interference, probably organics of some type. This supposition is supported by the TOC results from the water leach samples. The TOC values, although not high, are large enough to potentially interfere with fluoride and chloride detection. Further information regarding the IC analytes can be found in the full data packages (HASM 1993).

Table 5-21 shows the concentration of anions for each core composite sample. Similar levels for each of the analytes were found across all three core composites. Tables 5-22, 5-23, and 5-24 provide IC analyte concentrations as a function of depth. Tables 5-25, 5-26, and 5-27 provide additional information on other anions (such as total cyanide) as a function of depth. These anions were not determined by the IC method. Reported pH values for the subsegments and core composites are for 1:100 diluted samples; therefore, only the pH measurement of the liquid composite (direct pH measurements) is meaningful (Wodrich et al. 1992). The TOC and total inorganic carbon (TIC) assays are not considered capable of measuring the total cyanide in the waste because they depend on acid dissolutions to perform the analyses.

5.5.2 Liquid Core Composite

Except for F, no spike recoveries could be obtained for the anions on the liquid composite because sample concentrations exceeded spike concentrations by a factor of four or more. This behavior indicates various difficulties, such as matrix effects and low spike amounts relative to the sample concentrations. The poor fluoride spike recovery (750 percent) was affected by the low level of the analyte present, the presence of interferences (particularly organic anions), and the low spike levels. All of these factors make interpretation difficult and complicate quantitation of fluoride. Control standard recoveries range from 77 percent to 138 percent for all anions. The results of the liquid composite are consistent to the water-leached core composites and subsegments results, except for nitrate and nitrite. The nitrate and nitrite concentrations are approximately twice as high as those found in the solid samples. Total cyanide found in the sample was found to be unexpectedly high, approximately 5,400 ppm (Table 5-21).

Currently, there is no established explanation for this behavior. Although the sample was filtered, it is possibly the result of very small particulates (< $l\mu m$ diameter) in suspension in the liquid that were not filtered out prior to analysis.

5.5.3 Core 47

There are no spikes directly associated with the core composite material; the spike associated with Core 47-1B is used to verify analytical performance. The RPDs for the sample and duplicate runs for the core composite for each anion were generally excellent (< 5 percent), except for phosphate (18 percent), which was still within the 20 percent precision criteria. Spike recoveries ranged between 65 and 121 percent, indicating some minor matrix interferences. Samples exhibited characteristic poor spike recovery for fluoride (65 percent). Control standard recoveries ranged from 81 to 136 percent, indicating that the analysis was in control at the time of the assays. The RPDs for the subsegments were good, generally less than 10 percent, except for chloride and phosphate. However, the phosphate profile for Core 47 is very suggestive. The concentration increased significantly as a function of depth, from the 7,100 to 9,600 μ g/g range for subsegments 1B and 1C (with acceptable RPDs) to the 34,000 μ g/g and 55,000 $\mu g/g$ level for subsegment 1D. Even though the RPD for subsegment 47-1D is high, the results still indicate that the phosphate level is three to seven times higher at the bottom, indicating a different waste type. In addition, this trend is seen for PO_4^{5} in Cores 48 and 49. The general trend observed for all of the anions in this core is increasing concentration from top to bottom.

5.5.4 Core 48

There are no spikes directly associated with the core composite material; the spike associated with Core 48-1D is used to verify analytical performance. RPDs for the sample and duplicate runs for the core composite for each anion were generally good (< 15 percent), except for fluoride (138 percent) and phosphate (69 percent). This sample generally showed the highest RPDs across all anions, with few exceptions. This poor reproducibility of sample and duplicate is common for inorganic water leach assays on this sample matrix. The sample spike recoveries ranged from 38 percent to 126 percent. The behavior of the fluoride and phosphate spikes was attributed to sample inhomogeneity by the investigator, because 48-1D had high RPDs for these anions. The Core 48 subsegments showed slightly higher concentrations of several analytes than the other cores $(NO_2, NO_2, SO_4^2, and Cl^2)$. Control standard recoveries ranged from 81 percent to 133 percent.

5.5.5 Core 49

There are no spikes directly associated with the core composite material; the RPDs for the sample and duplicate runs for the core composite for each anion were generally good (< 15 percent). Subsegment 49-1C sample spikes were used to verify analytical performance. Spike recoveries for the anions ranged from 74 percent to 157 percent, indicating some matrix interferences. No

control standards were run with subsegment 49-1C. The RPDs for the subsegments were very good (< 10 percent) indicating sample homogeneity. Similar analyte profile behavior to Core 47 is observed, both in general and with regard to phosphate (the PO_4^{3-} RPD is better in this case).

Table 5-21. Anion Assays--Composite Data Results.

Analyte	Core 47 (µg/g)	Core 48 (µg/g)	Core 49 (μg/g)	Liquid composite (µg/g)
NO ₂	39,000	45,000	39,000	71,000
NO ₃	37,000	48,000	36,000	72,000
PO ₄ 3-	22,100	26,700	12,800	13,500
SO ₄ ²⁻	7,300	9,300	6,600	12,800
C1 ⁻	700	800	700	1,300
F	400	1,300	400	< 200
Free CN	820	1,300	550	1,340
Total carbon ¹	8,000	8,700	6,700	8,800
TOC ¹ ,	2,300	3,100	2,300	2,600
TIC ¹	5,700	5,600	4,400	6,200
Total cyanide ²	5,500	14,400	5,600	5,400
рН	10.75	10.08	9.37	12.08 ³

Total carbon, TOC, and TIC are not IC analyses, but are probably present as anions (TOC + TIC = Total carbon). Thus, it seems appropriate to include them with this table.

²Total cyanide is not an IC anion. Presently it is a developmental assay; however, the total cyanide assay is important in interpreting the data.

³Direct measurement.

IC = Ion chromatography $(NO_2^-, NO_3^+, PO_4^{3-}, SO_4^{2+}, C1^-, F^-, Free CN^-)$.

NM = No measurement.

TIC = Total inorganic carbon.

TOC = Total organic carbon.

NOTE: All IC results are obtained from a water leach preparation.

Table 5-22. Tank 241-C-109 Core 47 IC Analyte Trending.

Subsegment	NO ₂ - (μg/g)	NO ₃ (μg/g)	PO ₄ 3- (μg/g)	SO ₄ ²⁻ (μg/g)	Cl ⁻ (µg/g)	F- (μg/g)
1B	27,900	27,600	7,300	5,100	600	300
10	37,000	36,000	9,600	7,100	700	300
1D	40,000	38,500	44,500	7,400	800	300
Core comp.	39,000	37,000	22,100	7,300	700	400

Table 5-23. Tank 241-C-109 Core 48 IC Analyte Trending.

Subsegment	NO ₂ (μg/g)	NO ₃ - (μg/g)	PO ₄ 3- (μg/g)	S0 _ν ²⁻	Cl ⁻ (µg/g)	F [*] (μg/g)
10	51,000	56,000	15,800	11,000	. 950	500
1D	50,000	53,500	36,000	10,000	1,000	750
Core comp.	45,000	48,000	26,700	9,300	800	1,300

Table 5-24. Tank 241-C-109 Core 49 IC Analyte Trending.

Subsegment	NO ₂ (μg/g)	NO ₃ - (μg/g)	PO ₄ 3- (μg/g)	SO ₄ ²⁻ (μg/g)	C1 (µg/g)	F (μg/g)
1B	26,500	25,700	6,100	4,700	500	< 300
1C	44,000	42,000	8,800	8,200	800	300
1D	45,000	43,000	25,200	7,300	800	1,000
Core comp.	39,000	36,000	12,800	6,600	700	400

NOTE: All IC results are obtained from a water leach preparation.

Table 5-25. Tank 241-C-109 Core 47 Miscellaneous Analyte Trending.

Subsegment	TIC (µg/g)	TOC (µg/g)	Free CN ⁻ (μg/g)	Weight% Total carbon	рН
1B	5,400	2,200	570	0.76	8.82
1C	5,200	2,000	830	0.72	9.65
1D	5,400	2,200	910	0.76	10.21
Core comp.	5,700	2,300	820	0.80	10.75
Direct (Core comp.)	5,800	3,200	NA	0.90	NA

Table 5-26. Tank 241-C-109 Core 48 Miscellaneous Analyte Trending.

Subsegment	TIC (μg/g)	ΤΟC (μg/g)	.Free CN ⁻ (μg/g)	Weight% Total carbon	рН
1C	8,700	3,700	1,500	1.3	9.69
1D	7,500	3,500	1,400	1.1	10.99
Core comp.	5,600	3,100	1,300	0.87	10.08
Direct (Core comp.)	5,200	3,000	Not applicable	0.72	Not applicable

NA = Not applicable.
TIC = Total inorganic carbon.
TOC = Total organic carbon.
Direct = Measurement on a sample with little or no prior sample preparation.

Subsegment	TIC (μg/g)	ΤΟC (μg/g)	Free CN [*] (µg/g)	Weight% Total Carbon	рН
1B	3,900	1,800	370	0.57	10.22
1C	6,600	2,200	650	0.88	10.53
1D	6,800	2,600	720	0.94	10.95
Core comp.	4,400	2,300	550	0.67	9.37
Direct (Core comp.)	2,500	5,400	NA	0.79	NA

Table 5-27. Tank 241-C-109 Core 49 Miscellaneous Analyte Trending.

NA = Not applicable.

TIC = Total inorganic carbon.

TOC = Total organic carbon.

Direct = Measurement on a sample with little or no prior sample preparation.

5.6 ANALYTICAL RESULTS--RADIOCHEMISTRY

5.6.1 Radiochemistry Assays--General Comments

Analyses appear to be consistent. Total beta measurements calculated using 90 Sr detector efficiencies are largely in agreement with the sum of the major beta emitters, 90 SrY and 137 Cs. Similarly, the total alpha values show good agreement with the sum of the neptunium, plutonium, and americium/curium values. Detection of 137 Cs and most other radionuclides was observed to be a function of sample preparation. This was attributed to the ability of the sample preparation to dissolve the waste (KOH fusion dissolves the sample better than acid; acid dissolves the sample better than water). The GEA measurements are too low to show good agreement with alpha energy analysis for 241 Am. The GEA analytical values are back-corrected to January 1, 1992, to account for decay.

5.6.2 Gamma Energy Analysis Results

The GEA data from the replicate samples of the core composites and subsegments prepared by caustic fusion agree reasonably well (± 10 percent), with the exception of subsegment 49-1C. In general, the 137 Cs content is lowest in the 1B subsegments and highest in the 1D subsegments following the general trend observed for several analytes in this core (calcium, sodium, nickel, and PO_4^{3-}), increasing concentration from top to bottom. This behavior indicates that the 137 Cs is concentrated in the lower portion of the cores (and by extension, the tank). In addition, the total CN results directly trend the 137 Cs concentrations (i.e., high total CN = high 137 Cs measurement). The 154 Eu, 155 Eu, and 241 Am content is above the detection limits only in the 1B subsegments, indicating that these isotopes are in the upper waste levels of Cores 47 and 49. The GEA agreement between the sample and the

duplicates for the acid digestion core composites was not as good as that from the fusion samples (±20 percent). Water leach and liquid core composite results for ¹³Cs and total beta are two orders of magnitude lower than those obtained from fusion assays, indicating that the fission products are very insoluble. Agreement between the same top and bottom aliquots in the homogenization check samples (prepared by acid digestion) were not good, and differed in some cases by a factor of six, which indicates sample inhomogeneity or resistance to dissolution. The agreement in the homogenization check samples prepared by fusion was acceptable in each case.

5.6.3 Total Alpha Analysis and Uranium Assay

Total alpha, plutonium, and ²⁴¹Am analyses were performed on the fusion samples of the core composites and the direct filtered liquid composite. Total alpha measurements were also performed on the homogenization check samples from subsegments 48-1D and 49-1D and the water leach samples from all three cores. The total alpha activity was obtained by drying a small aliquot of fusion-prepared sample on a counting plate and determined using a scintillation detector. The plutonium and americium fractions were separated by solvent extraction or ion exchange and similarly counted.

The plutonium analyses are reported as total alpha plutonium. The process blank was two to three orders of magnitude lower that the samples, indicating little contamination occurred during sample preparation. Because of the low total alpha concentration for the liquid composite, no specific nuclide analyses were performed. The total alpha concentration tends to be somewhat lower than the sum of the individual alpha emitters; the difference is likely because of absorption by the salt residue on the counting mounts. Isotopic resolution of the samples was obtained from thermal ionization mass spectroscopy. Because the ²³⁸Pu concentration was low and the ²³⁸U concentration was relatively high in the core composites, the uranium contamination interfered with the determination of ²³⁸Pu, thus that plutonium isotope was determined from alpha energy analysis. The variation in the plutonium isotopic composition is noteworthy, especially for ²⁴⁰Pu. Sample and duplicate analyses are generally within acceptable limits.

Uranium measurements were obtained from ICP fusion and laser fluorimetry of the three core composites and the liquid composite core. The assays show good agreement between duplicates, and reasonably good agreement between all three cores for both assays. Core 48 again demonstrated the highest RPD (18 percent). The liquid composite had a much lower uranium content than any of the solid core composites. This result is not surprising; because of the alkaline environment of the tanks, the uranium is likely a highly insoluble precipitate. The uranium content varies by a factor of four between the three cores, with Core 48 having the highest concentration and Core 49 having the lowest. There is no observable general trend of the uranium concentration as a function of depth.

5.6.4 Total Beta

Total beta, ⁹⁰Sr, and ⁹⁹Tc analyses were performed on the water leach and fusion samples of the core composites and the direct filtered liquid

composite. Strontium-90 measurements were also performed on the fusion prepared samples from all of the subsegments. The total beta activity was determined by drying a small aliquot of prepared sample on a beta proportional counter. The 90Sr and 97Tc fractions were separated by solvent extraction or ion exchange and similarly counted. There are generally acceptable levels of agreement between replicates. Preparation blank beta activities for these samples are orders of magnitude lower than the levels found in the samples, again indicating little contamination from preparation in the hot cell.

Most of the beta activity in the tank liquid composite and water leaches of the solid core composites is from ^{137}Cs , except for the Core 49 water leach composite sample, where ^{90}SrY is the dominant beta emitter. The ^{90}Sr data is consistent between the fusion core composite values and the subsegments from that core. The ^{90}Sr content is much higher in subsegments 47-1B and 49-1B the in the lower cores, and the general trend of the ^{90}Sr concentration is lower as a function of depth.

Tables 5-28, 5-29, 5-30, 5-31, 5-32, 5-33, and 5-34 show the radionuclide concentrations found in the core composite samples. Tables 5-35, 5-36, and 5-37 show fission product concentration and uranium concentrations as a function of depth.

Table 5-28.	Core	Composite	Fission	Products	(Fusion	Prep).
-------------	------	-----------	---------	----------	---------	--------

Core No.	¹³⁷ Cs (μCi/g)	⁹⁰ Sr. (μCi/g)	154Eu (μCi/g)	¹⁵⁵ Eu (μCi/g)	⁶⁰ Co (μCi/g)	Total beta (μCi/g)
Core 47	870	1,180	< 0.24	< 0.86	< 1.4 E-02	2,750
Core 48	1,030	190	< 0.73	< 1.20	< 2.7 E-02	1,300
Core 49	560	930	0.36	< 0.52	< 1.4 E-02	2,300

Table 5-29. Liquid Core Composite Fission Products (Acid Prep).

Core No.	¹³⁷ Cs (μCi/g)	⁹⁰ Sr (μCi/g)	¹⁵⁴ Eu (μCi/g)	¹⁵⁵ Eu (μCi/g)	⁶⁰ Co (μCi/g)	Total beta (μCi/g)
Liquid composite	5.61	9.9E-02	< 3.0E-04	< 2.4E-03	1.46E-03	5.43

Table	5-30.	Core	Composite	Uranium.
-------	-------	------	-----------	----------

Core No.	U _{ICP} fusion (μg/g)	υ _{ει} (μg/g)	238 _U Mass fraction	²³⁵ U Mass fraction
Liquid composite	< DL	3.7	, NM	NM
Core 47	9,200	12,000	0.993263	0.006573
Core 48	24,700	27,600	0.993038	0.006852
Core 49	4,700	7,500	0.993109	0.006753

< DL = Below detection limits.

FL = Uranium measurement by laser fluorimetry.

Table 5-31. Core Composite Trace Radionuclides.

Core No.	⁹⁹ Tc (μCi/g)	³ H** (μCi/g)	¹⁴ C (μCi/g)
Liquid composite*	1.6E-01	3.0E-03	2.3E-03
Core 47	1.1E-01***	8.5E-03	6.3E-06
Core 48	1.2E-01***	6.4E-03	1.8E-05
Core 49	9.4E-02***	6.4E-03	3.6E-05

*Liquid composite results obtained from acid digestion. ** A values are biased high from contamination in the hot

cell (blank levels ranged from 3.9 E-03 to 6.7 E-03).

*** Tc core composite values obtained from caustic fusion assay; 3H and 14C values obtained from water digestion.

Table 5-32. Plutonium Concentration and Isotopic Distribution.

Core No.	Total plutonium α activity (μCi/g)	²³⁸ Pu Mass fraction	²³⁹ Pu Mass fraction	²⁴⁰ Pu Mass fraction	²⁴¹ Pu Mass fraction	²⁴² Pu Mass fraction
Core 47	0.88	0.00005	0.932237	0.066256	0.001216	0.000241
Core 48	0.065	0.00011	0.976356	0.022995	0.000364	0.000176
Core 49	0.079	0.00014	0.949629	0.048786	0.001119	0.000329

No Pu measurement on liquid composite, concentration too low.

NM = No isotopic measurement on liquid composite, concentration too low.

		•			` '	•	,
Core No.	²³⁷ Np (μCi/g)	²³⁸ Pu* (μCi/g)	²³⁹ Pu* (μCi/g)	²⁴¹ Am _{GEA} (μCi/g)	²⁴¹ Am _{AEA} (μCi/g)	Total α (μCi/g)	Total α water leach (μCi/g)
Core 47	3.65E-04	4.40E-05	0.82	< 0.58	0.32	0.992	4.84E-03
Core 48	3.34E-04	7.15E-06	0.063	< 0.71	0.01	0.065	1.35E-04
Core 49	3.01E-04	1.11E-05	0.075	< 0.35	0.13	0.129	6.25E-04

Table 5-33. Core Composite Transuranics (fusion preparation).

Table 5-34. Total Alpha Homogenization Test (μ Ci/g) (Acid Prep).

	Top-1	6.18E-02	Top-2	5.00E-02
Core 48-1D	Bottom-1	6.74E-02	Bottom-2	5.26E-02
	Top-1	3.58E-02	Top-2	5.94E-02
Core 49-1D	Bottom-I	4.46E-02	Bottom-2	5.23E-02

Table 5-35. Tank 241-C-109 Core 47 Radionuclide Trending (Fusion Preparation).

Subsegment	¹³⁷ Cs (μCi/g)	⁹⁰ Sr (μCi/g)	154 Eu (μCi/g)	¹⁵⁵ Eu (μCi/g)	²⁴¹ Am (μCi/g)	υ _{ις} (μg/g)
1B	340	4,600	0.88	1.16	0.75	11,800
1C	770	470	< 0.13	< 0.86	< 0.48	6,100
1D	950	200	< 0.11	< 1.02	< 0.55	5,800
Core comp.	870	1,180	< 0.24	< 0.86	< 0.58	9,200
Core comp. (water leach)	9.24	17.35*	< 0.002	< 0.008	< 0.004	< DL

^{*}Value from total beta analysis.

^{* =} Determined from total plutonium alpha and isotopic measurements

AEA = Measurement by alpha energy analysis.

GEA = Measurement by gamma energy analysis.

ICP = inductively coupled plasma result.

		•	•			
Subsegment	¹³⁷ Cs (μCi/g)	⁹⁰ Sr (μCi/g)	¹⁵⁴ Eu (μCi/g)	¹⁵⁵ Eu (μC1/g)	²⁴¹ Am _{GEA} (μCi/g)	υ _{ιςΡ} (μg/g)
10	1,200	150	< 0.08	< 1.10	< 0.59	16,700
10	1,170	120	< 0.10	< 1.20	< 0.63	14,400
Core comp.	1,030	190	< 0.73	< 1.2	< 0.71	24,700
Core comp. (water leach)	9.3	8.6*	< 0.0007	< 0.008	< 0.004	< DL

Table 5-36. Tank 241-C-109 Core 48 Radionuclide Trending (Fusion Preparation).

Table 5-37. Tank 241-C-109 Core 49 Radionuclide Trending (Fusion Preparation).

Subsegment	¹³⁷ Cs (μCi/g)	⁹⁰ Sr (μCi/g)	¹⁵⁴ Eu (μCi/g)	¹⁵⁵ Eu (μCi/g)	²⁴¹ Am _{GEA} (μCi/g)	U _{icp} (μg/g)
18	120	2,400	0.78	0.93	0.52	7,900
1C	350	200	< 0.04	< 0.25	< 0.14	1,300
1D	700	190	< 0.06	< 0.50	< 0.27	12,400
Core comp.	560	930	0.36	< 0.52	< 0.35	4,700
Core comp. (water leach)	5.3	8.7*	<0.0020	< 0.0060	< 0.003	< DL

^{*}Value from total beta analysis.

5.7 ANALYTICAL RESULTS—ENERGETICS

Scanning TGA and DSC were performed on subsegment and core composite material obtained from tank 241-C-109. These two thermal analysis techniques are useful in determining the thermal stability or reactivity of a material. In DSC analysis, heat flow over and above the usual heat capacity of the substance is measured while the substance is exposed to a linear increase in temperature, i.e., dT/dt = Constant (where T = temperature, and t = time). While the substance is being heated, air is passed over the waste material to remove any gases being released. The onset temperature for an endothermic or exothermic event on a DSC is determined graphically.

TGA measures the mass of a sample while the temperature of the sample is increased at a constant rate (5 $^{\circ}$ C/min). Again, dT/dt is constant because the X-axis is representative of the running time of the analysis as well as the temperature increase of the sample during analysis. The Y-axis represents the weight percent of the sample and is effectively unitless. As with the DSC,

< DL = below detection limit.

ICP = inductively coupled plasma result.

air is passed over the sample during heating. Any decrease in the weight percent of the sample represents a loss of gaseous matter from the sample either through evaporation or through a reaction that forms gas phase products.

DSC is often used to measure thermal decomposition temperatures, heats of reaction, reaction temperatures, melting points, and solid-solid transition temperatures. TGA is used to measure thermal decomposition temperatures, water content, and reaction temperatures. The two methods often provide complementary information.

5.7.1 Remarks on the Interpretation of Differential Scanning Calorimetry/Thermogravimetric Analysis Data

The results of the thermal analyses performed are summarized in Tables 5-38 and 5-39. The observed behavior for each of the samples is similar; there are two significant features on the DSC trace; there are also three distinguishable features on the TGA plots. Because one of the DSC events overlaps the area where two of the TGA events are occurring, there may be more phenomena occurring in this temperature range than the machine is able to resolve clearly. The values presented in the tables do not exactly match the values recorded on the DSC and TGA plots. This is because interpreting these semi-quantitative analyses requires considerable experience and judgement on the part of the analyst. Although the temperature ranges observed for the various transitions in the DSC and TGA assays do not exactly match, the weight losses and thermal events in the observed transitions are considered related and usually in the same vicinity.

There is a concern regarding the choice of cover gas affecting the DSC and TGA results. Air was used in the assays instead of an inert gas because that is what the test instructions directed. However, oxygen in the air may contribute to the oxidation of the sample and alter the reaction (Pederson et al. 1993). This condition is not considered representative of the potential reaction conditions in the tank; therefore, future DSC/TGA tests will be performed under an inert cover gas. Test instructions governing the treatment of these samples are being written and implemented to ensure the proper thermal testing of ferrocyanide waste samples.

Thermogravimetric Analysis Results from Tank 241-C-109. Table 5-38.

Core sample	Total Wt% loss	Transition 1 Wt% loss	Transition 2 Wt% loss	Transition 3 Wt% loss
47-1B	31.4	10.2	17.9	3.3
47-1C	39.3	18.0	17.6	3.7
47-1D	28.2	19.7	6.8	1.7
47-Comp.	33.4	14.8	14.9	3.7
48-1C	N.M.	N.M.	N.M.	N.M.
48-1D	48.1	45.1	3.2	-0.2
48-Comp.	NM	NM ·	NM	. NM
49-1B	34.1	4.2	25.8	4.1
49-1C	46.6	29.6	14.2	2.8
49-1D	40.0	29.3	9.6	1.1
49-Comp.	46.1	26.6	15.8	3.7

Transition 1: 31° - 150 °C.
Transition 2: 150° - 425 °C.
Transition 3: 330° - 500 °C.

These ranges are appropriate and there is some overlap.

NM = No measurement.

Comp. = Core composite.

Table 5-39. Differential Scanning Calorimetry Energetics Results from Tank 241-C-109.

	Transition 1			Tra	nsition	Tran	Transition 3		
Core Sample	Range (°C)	Avg. onset (°C)	Avg. ∆H (J/g)	Range (°C)	Avg. onset (°C)	Avg. ΔΗ (J/g)	Range (°C)	Avg. onset (°C)	Avg. ΔH (J/g)
47-1B	33-150	70	350	190-338	259	1,555	(a)	N.A.	
47-1C	35-144	53	425	167-318	217	610	380-461	391	72
47-1D	34-154	59	767	190-369	225	508	369-441	375	21
47-Comp.	34-150	55	785	159-330	216	1,084	(a)	N.A.	
48-1D	34-196	104	1,034	249-338	272	-27	336-431	359	31
49-1B	33-115	40	368	193-373	270	2,188	(a)	N.A.	
49-1C	33-197	72	658	167-316	242	565	(a)	N.A.	
49-1D	34-166	71	712	152-324	225	305	379-483	394	48
49-Comp.	34-192	99	964	190-329	243	922	(a)	N.A.	

(a) No quantifiable transition is observed.
 NOTE: To convert from J to cal, divide by 4.18.
 NOTE: Negative ΔH indicates an exotherm.
 N.A. = Not applicable.
 Comp. = Core composite.

5.7.2 General Comments on the Differential Scanning Calorimetry/Thermogravimetric Analysis Behavior of the Samples

The first transition in each sample is endothermic, begins at the lower temperature limit of the analysis (30 °C), and is essentially complete between 140 °C and 200 °C. The most likely phenomenon occurring in this region are the release of the bulk and interstitial water in the core sample material. The endotherms exhibited in this region are substantial (typically, 350 to 1,030 J/g). These values are per gram of wet sample; if divided by the mass fraction lost during analysis, they typically range from 2,200 to 3,900 J/g and correspond generally with the heat of vaporization of water (2,260 J/g), although there are some outliers with much higher endotherms. The overall TGA water content does not correspond well with the water loss observed in a gravimetric weight percent solids determination as given in Tables 5-43 to 5-45. However, this disagreement is attributed to phenomenological differences in the materials' reaction to thermal stress of varying intensity.

Additional weight loss and endotherms are routinely detected between 260° to 300°C in the Core 47 and 49 samples. For these samples, the majority of the weight percent change observed in the TGA curve occurs over this temperature range and no exothermic action is observed. This behavior is attributed to the high levels of aluminum in the samples. It is believed that the phenomenon occurring in this region is the dehydration of aluminum hydroxide to alumina and water vapor (Brown and Jensen 1993; Appendix A). Several other reactions are potentially associated with this endotherm: melting of NaNO2 and NaNO3 salts, endothermic ferrocyanide reactions with the nitrate and nitrite salts to form NO and NO, and water losses of sodium alumina silicates and other hydrated compounds. Core 48 is unlike the other samples and exhibits exotherms and weight changes that have been observed in previous thermal analysis studies of tank 241-C-112 waste (Simpson et al. 1993), Cs, NiFe(CN), (Scheele et al. 1991) and other simulant materials (Bechtold 1992; Jeppson 1993). As reported previously, the dried simulant materials demonstrate much larger exothermic responses than those observed in tank 241-C-109 waste. However, the magnitude of the exotherms observed correlates roughly with the predicted exotherms derived from the amount of cyanide present in the waste (refer to Tables 5-40, 5-41 and 5-42), based on the Fauske (1992) determined value of -3.95 kJ/g Na2NiFe(CN)6. The weight losses are attributed to the loss of gaseous reaction products and waters of hydration.

The third transition is very small compared with the other two observed transitions (< 100 J/g), but here the energetic behavior is not readily quantifiable in all of the samples analyzed. A minor weight loss was routinely observed in the samples at temperatures above 300 °C.

			·			
Subsegment	Wt% Total cyanide (dry)	Equivalent Wt% sodium nickel ferrocyanide (dry)	Theoretical heat of reaction* (cal/g dry waste)	Measured heat of reaction** (cal/g dry waste)		
1B	0.30	0.61	-5.8	No Exotherm		
1C	0.44	0.89	-8.4	No Exotherm		
1D	0.58	1.17	-11.1	No Exotherm		
Composite	0.55	1.11	-10.5	No Exotherm		

Table 5-40. Tank 241-C-109 Core 47 Energetic Comparison.

Table 5-41. Tank 241-C-109 Core 48 Energetic Comparison.

Subsegment	Wt% Total cyanide (dry)	Equivalent Wt% sodium nickel ferrocyanide (dry)	Theoretical heat of reaction* (cal/g dry waste)	Measured heat of reaction (cal/g dry waste)	
1C	1.13	2.29	-21.6	NM	
1D	0.87	1.76	-16.6	-12.4	
Composite	1.44	2.91	-27.5	NM	

Table 5-42. Tank 241-C-109 Core 49 Energetic Comparison.

Subsegment	Wt% Total cyanide (dry)	Equivalent Wt% sodium nickel ferrocyanide (dry)	Theoretical heat of reaction* (cal/g dry waste)	Measured heat of reaction** (cal/g dry waste)
18	0.35	0.71	-6.7	No Exotherm
1C	0.81	1.64	-15.5	No Exotherm
1D	0.55	1.11	-10.5	No Exotherm
Composite	0.56	1.13	-10.7	No Exotherm

NM = No measurement.

NOTE: 1 cal = 4.18 J.

The properties related to energetics are illustrated for each core in Tables 5-43, 5-44, and 5-45. The results for the samples from 48-1D, indicates that this sample differs in thermal behavior from most of the other samples, further suggesting a difference in waste type.

^{*}Based on -3.95 kJ/g Na₂NiFe(CN)₆ (Fauske 1992).
**Endothermic measurements are in Appendix A, Table A-9.

Subsegment	Wt% Total cyanide (dry)	Wt% Total organic carbon	Wt% Total carbon	Wt% Water (Grav.)	Wt% Water (TGA)	Average heat of reaction (J/g dry waste)
1B	0.30	0.22	0.76	19.3	31.4	No Exotherm
1C	0.44	0.20	0.72	28.4	39.3	No Exotherm
1D	0.58	0.22	0.76	39.4	28.2	No Exotherm
Composite	0.55	0.23	0.80	21.5	33.4	No Exotherm

Table 5-43. Tank 241-C-109 Core 47 Energetics Trending.

Table 5-44. Tank 241-C-109 Core 48 Energetics Trending.

Subsegment	Wt% Total cyanide (dry)	Wt% Total organic carbon	Wt% Total carbon	Wt% Water (Grav.)	Wt% Water (TGA)	Average heat of reaction (J/g dry waste)
1C	1.13	0.37	1.24	52.8	NM	NM
1D	0.87	0.35	1.10	51.6	48.1	-51.9
Composite	1.44	0.31	0.87	57.7	NM	NM

Table 5-45. Tank 241-C-109 Core 49 Energetics Trending.

Subsegment	Wt% Total cyanide (dry)	Wt% Total organic carbon	Wt% Total carbon	Wt% Water (Grav.)	Wt% Water (TGA)	Average heat of reaction (J/g dry waste)
18	0.35	0.18	0.57	19.6	34.1	No Exotherm
10	0.81	0.22	0.88	38.3	46.6	No Exotherm
1D	0.55	0.26	0.94	39.6	40.0	No Exotherm
Composite	0.56	0.23	0.67	27.8	46.1	No Exotherm

Heats of Reaction are calculated using the TGA wt% water value.

NOTE: 1 cal = 4.18 J.

Grav. = Water content from gravimetric weight percent water.

NM = Not measured.

TGA = Water content from scanning thermogravimetric analysis.

The TOC and TIC assays are not considered capable of measuring the total cyanide in the waste because they depend on acid dissolutions to perform the analyses.

5.8 ANALYTICAL RESULTS - POTENTIAL WASTE CONSTITUENTS

5.8.1 Mass Balances

A method to help ensure that the data are acceptable is to perform a mass balance on the core composite sample data. This activity functions as a rough quality control check, and also provides insight to some of the properties of the matrix. To do this, the assumption in performing the mass balance is that the anions, cations, and water are all associated in some manner, but the exact chemistry of the association is not considered. Analytes contributing less than 0.2 wt% (generally trace ICP analytes, AA analytes, and radionuclides) are considered negligible in this assessment. The assays that contributed analytes to the mass balance were the ICP fusion, IC, total carbon, total cyanide assays, and the gravimetric wt% water measurement. The ICP fusion value does not include nickel, which is a significant analyte in the sample but may be biased high. However, for the purpose of this exercise, the nickel value from the respective acid leach preparations will be inserted into the total mass of ICP fusion analytes to account for it.

Without considering the physical and chemical properties of the waste matrix and the context of the process history, the mass balances produced from these assays will be biased low. However, this bias is expected because it is known that there are analytes present that were not measured in the analysis of the samples. While the IC anion analysis only measures the water-soluble components; there is a substantial insoluble residue that must contain additional anions. There is no measurement of the sulfide content in any of the assays, even though it has been previously established that 21,600 g-mol of S-2 was used in scavenging ^{60}Co . Thus, an additional contribution of 2,300 μg S²-/g has been estimated as necessary to close the balance. Bismuth was not reported in the assays, and BiPO4 first cycle waste was recorded as being disposed here, which also introduces a potential shortfall.

Aluminum is likely to be present as $Al(OH)_3$, and other transition metals are also likely to be present as hydroxides or hydrous metal oxides. Neither hydroxide ion or oxide content has been measured in the waste, which introduces additional sources of shortfall in the recovery. Therefore, multipliers for aluminum (2.9), iron (1.6), nickel (1.6), and uranium (1.3)will be used to account for the unmeasured hydroxide or oxygen, which are assumed to be present in combination with these analytes (Appendix A). Only metals making weight percent contributions to the waste matrix will be adjusted in this manner; the trace metals will be assumed to be lost in the error of the major constituents. Adjustments will be made individually for TOC, TIC, and total cyanide. It is assumed that the TOC and TIC assays did not consume or measure any cyanide present. In addition, a significant disparity can be corrected by comparing the soluble phosphorus from the water leach ICP (and assuming that it is $P0_4^{32}$), $P0_4^{32}$ values from the IC, and the phosphorus from the ICP fusion assay; the phosphate was found to be only 29 to 43 percent soluble (Appendix A). The water leach ICP and IC values agree within 3 percent, strongly suggesting that the soluble phosphorus in the waste matrix is present as PO₄. The process history of the tank also indicates that large amounts of phosphate were used to encourage precipitate formation. Therefore, an assumption that the phosphorus in the fusion assay represents an insoluble PO_4^{5-} is not unwarranted. Convert the phosphorus in the ICP assay

to PO_4^{3-} and add it along with the water soluble phosphate and other anions. A minor accounting shift is now necessary to avoid double counting (subtract the ICP fusion phosphorous value).

Accounting for the analytes in this manner aids in closing agreement and the percent recoveries are between 96.3 percent and 106.1 percent (near quantitative recoveries). However, there remain some aspects of the waste matrices that require examination. Tables 5-46, 5-47, and 5-48 present mass balances that have been adjusted to compensate for the contributions of unmeasured (but likely) analytes combined with the measured analytes. There may have been some error introduced from drying of the sample during the preparation of the core composite. In the case of these waste materials, the disparity between the gravimetric water measurement and the TGA water content suggests (1) drying of the sample before the gravimetric assay; (2) incomplete drying during the gravimetric test, which biases the results low; or (3) One or more endothermic events occurring at the same time (chemical reactions or phase transitions resulting in the loss of mass).

Table 5-46. Adjusted Mass Balance: Core 47 Composite.

Assay	Concentration (#g/g)
ICPFusion (+ Ni from acid leach; -P; Al, Fe, Ni, U, adjustments)	567,600
IC Anions (TOC, TIC, and CN adjustments; +P as PO ₄ +S ²)	212,500
Gravimetric Water	215,000
Total (1,000,000 μg/g)	995,100

Table 5-47. Adjusted Mass Balance: Core 48 Composite.

Assay	Concentration (µg/g)
<pre>ICPFusion (+ Ni from acid leach; -P; Al, Fe, Ni, U, Si adjustments)</pre>	241,500
IC Anions (TOC, TIC, and CN adjustments; +P as PO ₄ +S ²)	242,600
Gravimetric Water	577,000
Total (1,000,000 μg/g)	1,061,100

IC = Ion chromatography.

ICP = Inductively coupled plasma spectroscopy.

TIC = Total inorganic carbon.

TOC = Total organic carbon.

Table 5-48. Adjusted Mass Balance: Core 49 Composite.

Assay	Concentration (µg/g)
<pre>ICPFusion (+ Ni from acid leach; -P; Al, Fe, Ni, U, adjustments)</pre>	503,700
IC Anions (TOC, TIC, CN adjustments; +P as PO ₄ ³⁻ ; +S ²⁻)	181,600
Gravimetric water	278,000
Total (1,000,000 μg/g)	963,300

IC = Ion chromatography.

ICP = Inductively coupled plasma spectroscopy.

TIC = Total inorganic carbon.

TOC = Total organic carbon.

5.8.2 Suggested Components of Waste Matrix

The actual composition of the waste matrix is quite complex, and trace amounts of various compounds probably exist in the tank. However, with some simple assumptions regarding how the anions and cations will combine, a list of the most probable compounds that exist in the waste matrix and contribute significantly to its overall makeup can be developed.

Table 5-49 is a condensed version of a more general chart found on page D-147 in the Handbook of Chemistry and Physics 64^{th} Ed. (Weast 1984). It provides solubility data on some of the most common anions and cations.

Table 5-49. Probable Solids in the Waste Matrix.

	NO ₂	NO ₃	PO ₄ 3-	SO ₄ 2-	OH-	CN ⁻ (as Fe(CN) ₆ ⁴⁻	CO ₃ ²⁻	S ²⁻
A1 ⁺³			PPT		PPT	PPT		NL
Ca ⁺²			PPT	PPT			PPT	
Fe ^{+2,+3}					PPT	PPT	PPT	PPT
Na⁺								
Ni ⁺²			PPT		PPT	PPT	PPT	PPT
U ⁺⁶		NL	PPT	PPT	PPT			PPT

PPT = Precipitate forms.

NL = Precipitate formation not likely under tank conditions.

From the earlier tables and process information, chloride, sulfide, and even ferrocyanide will not be significant mass contributors to the waste matrix. Sulfide and cyanide precipitates are significant because they provide a potential fuel source. However, it is generally believed that the sulfides were eventually converted to sulfates. Sodium, NO_2 , and NO_3 are highly soluble, and thus probably do not contribute much to the insoluble solids. However, sodium, nitrate, and nitrite contribute significantly to the overall solids content of the waste (dissolved + insoluble solids). In addition, they represent three of the four most prevalent analytes, after water, in the waste. No analytical measurement of hydroxide was made, but it is known that in the process_history of tank 241-C-109, basic solutions were added routinely to the tank. The following is a list of likely candidates for the insoluble solids.

- Aluminum hydroxide, Al(OH),
- Aluminum phosphate, AlPO₄

Aluminum oxide, Al₂0₃

- Aluminum silicate, 3Al₂0₃•2Si0₂
- Tetraaluminum ferrocyanide, AT₄[Fe(CN)₆]₃
- Calcium phosphate, $Ca_3(PO_4)_2$
- Calcium sulfate, CaSO,
- Calcium carbonate, CaCO₃
- Iron carbonate, FeCO_z
- Iron(II) hydroxide, Fe(OH),
- Iron(III) hydroxide, Fe(OH)₃
- Iron(II) phosphate, $Fe_3(PO_4)_2$
- Iron(III) phosphate, FePO₄
- Iron sulfide, FeS
- Iron(III) ferrocyanide, Fe₄[Fe(CN)₆]₃
- Disodium nickel ferrocyanide, Na, NiFe(CN),
- Nickel carbonate, NiCOz
- Nickel sulfide, NiS
- Nickel hydroxide, Ni(OH),
- Dinickel ferrocyanide, Ni₂Fe(CN)₆
- Uranyl phosphate, UO2HPO4•4H2O Uranyl hydroxide, UO2(OH)2
- Uranyl sulfide, UO,S
- Uranyl sulfate, $2(\overline{U}0_{2}SO_{4}) \cdot 7H_{2}O$.

The 137Cs present is still apparently bound with the ferrocyanide, and the 90Sr is probably bound with phosphate, carbonate, or sulfate.

5.8.3 Comparison to Theoretical Estimates and Simulant Studies

Agreement between synthetic sludge properties and observed waste material characteristics is within the constraints of the synthetic recipes and assumptions regarding chemical behavior in tank 241-C-109. Table 5-50 compares some properties and analyte concentrations of the waste materials and comparable simulants.

Analyte	In Farm 2 simulant values	Core 47 values subsegment range (composite value)	Core 48 values subsegment range (composite value)	Core 49 values subsegment range (composite value)
Ni ug/g*	18,700	19,400 to 25,600 (31,900)	44,000 to 24,000 (33,000)	10,800 to 31,900 (22,900)
Wt% H ₂ O (Grav.)	51	19.3 to 39.4 (21.5)	51.6 to 52.8 (57.7)	34.1 to 46.6 (46.1)
Wt% Total Cyanide dry basis	9.1 to 11.3	0.30 to 0.58 (0.55)	0.87 to 1.13 (1.44)	0.35 to 0.81 (0.56)
ΔH J/dry g	-1,200	No detectable exotherm	-51.9 (NM)	No detectable exotherm
Density g/ml	1.39**	NM (1.2)	NM (1.3)	NM (1.1)

Table 5-50. Tank 241-C-109 Comparison of Waste Material with Simulants for Selected Analytes.

*Nickel analysis is biased high. Values are derived from ICP fusion performed in a nickel crucible. However, in each case the blank value was an order of magnitude (or greater) less than the measurement. The fusion values are usually twice the acid digestion values, for the core composite measurements.

**Centrifuged for 30 gravity years.

Grav. = gravimetric assay.

NM = Not measured.

5.9 RCRA-TYPE ANALYSIS: DATA VALIDATION/VERIFICATION PROTOCOL

5.9.1 Chemical Data

Data validation procedures for chemical data were in place during the analysis of tank 241-C-109. The data validation and verification procedures followed to ensure reliable data for RCRA-type samples are described in detail in WHC-CM-5-3, Sample Management and Administration, Section 2.0. A brief list of the requirements for data packages are as follows:

- Requested versus reported analyses
- Analysis holding times
- Matrix spike/matrix spike duplicate analysis
- Surrogate recoveries
- Duplicate analysis
- Analytical blank analysis
- Additional QA/QC oversight, as designated in the SOW
- Initial and continuing instrument calibration
- GC/MS Tune criteria (GC/MS analysis)

- Internal standards (GC analysis)
- Laboratory control samples (LCS)
- Interference check sample (ICP).

When determining the quality of the chemical data for tank 241-C-109, it is useful to consider the results of several of the quality control assays. Blanks, matrix spikes, duplicates, and control samples can all provide further insight to the data and its reliability. Potential sample contamination problems are addressed using analytical blanks. Confounding effects of the sample matrix are resolved using matrix spike results. Duplicate analysis compares the difference between the replicate samples, providing an indication of laboratory precision (and in some cases, sample heterogeneity). The laboratory control sample offers a monitor of overall performance of an analytical method in all steps of the analysis. Overall, there were few problems with the data validation and compliance with established quality control criteria. The 241-C-109 samples were generally free from calibration and contamination errors. In addition, the interference control standards, matrix spike, laboratory control standard, and holding time requirements were largely met. In some cases, the poor sample/duplicate agreement observed in some of the assays was attributed to significant sample heterogeneity. In almost all cases, no significant problems were found, and the data obtained was qualifiable and usable in characterizing the tank contents. Appendix D contains a summary of the most relevant quality assurance data.

5.9.2 Radiological Data

Similarly, the data validation and verification procedures followed to ensure reliable data for radioactive, high-level, RCRA-type samples are also described in detail in WHC-CM-5-3, Sample Management and Administration, Section 2.4. They differ somewhat from the requirements for chemical data. A brief list of the requirements for data packages are as follows:

- · Chain of custody
- Requested versus reported analyses
- Efficiency checks
- Laboratory control samples (LCS)
- Initial calibration
- Preparation blank analysis
- Matrix spikes/tracers/carriers
- Additional QA/QC oversight, as designated in the SOW
- Duplicate analysis
- Background checks.

When determining the quality of the radiological data for tank 241-C-109, it is also useful to consider the results of several of the quality control assays. Chain-of-custody, calibrations, efficiency and background checks, blanks, matrix spikes/tracers/carriers, duplicate analyses, and laboratory control samples can all provide further insight to the data and its reliability. Potential sample contamination problems or loss of sample control are addressed in using a chain-of-custody. Initial calibrations, efficiency and background checks, and analytical blanks ensure that the equipment is operating correctly and further address contamination problems. Confounding effects of the sample matrix are resolved using matrix

spike/tracers/carriers. Duplicate analysis compares the difference between the replicate samples, providing an indication of laboratory precision (and in some cases, sample heterogeneity). The laboratory control sample offers a monitor of overall performance of an analytical method in all steps of the analysis. In the case of the radiological data, there were several problems with the data validation and compliance with established quality control criteria. The 241-C-109 chain-of-custody documentation showed liner liquid apparently came from two samples (Cores 47 and 49) thus, sample integrity was compromised. With the degree of sample containment and isolation these samples have, there was no danger of significant sample contamination or excessive exposure risk. However, this incident does demonstrate some shortcomings of the present sampler. The sample blank results further indicated that samples were generally free from contamination errors (tritium was an exception). Additionally, there are several problems in compliance with the established QC criteria for initial calibrations, efficiency checks, matrix spike/tracers/carriers, and the use of laboratory control standards for these samples. In many cases, the radiological data obtained was determined to be not qualifiable and unusable in characterizing the tank contents during the validation procedure. On further investigation, it was found that this finding is true only because of the discrepancies in quality control criteria between the PNL Technical Task Plan and the governing validation documentation WHC-EP-0210 (Winters et al. 1990). These concerns were addressed and responded to in the PNL TTP, which outlined the alternative quality control criteria that would be adhered to during the analysis of the samples. WHC agreed to the criteria outlined in the PNL TTP and a formal audit response has been issued to clarify the matter further (Appendix D). For the purposes of characterization and data interpretation stated in this document, the data are deemed acceptable for use.

6.0 INTERPRETATION OF ANALYTICAL RESULTS

6.1 TANK 241-C-109 WASTE PROFILE

Tank 241-C-109 received several major types of waste likely to deposit solids during its operating history. The waste types in chronological order are as follows:

- Bismuth phosphate first decontamination cycle waste (1C)
- Unscavenged uranium recovery waste from U Plant and ferrocyanidescavenged waste (FeCN) from scavenging of UR waste initially stored in other tanks
- Ferrocyanide-scavenged supernatants from BY Tank Farm that required additional scavenging for 60Co and 90Sr without ferrocyanide
- Ferrocyanide-scavenged waste (FeCN) 1C Evaporator Bottoms (EB) from scavenging of evaporator processed 1C, CW, and UR waste initially stored in other tanks
- Decladding/coating (CW) and hot semiworks (HS) wastes.

A relatively large volume of B Plant ion-exchange waste was received after these solids-bearing wastes. These last wastes would not be expected to contribute large amounts of solids to the tank. This section will attempt to identify the location of the tank waste solids, thereby allowing estimates of the tank inventory for various analytes of importance.

To identify the waste profile, the approach taken was that the subsegment assays were examined for analytes distinct to the waste types disposed in the tank, and that information was combined with what is known regarding the tank's process history. The first waste placed in the tank, via the cascade inlet from tank 241-C-108 was BiPO₄ 1C waste. This waste would be comparatively high in bismuth, phosphate, and aluminum because aluminum decladding waste was combined with it. The 1C solids volume was measured as 38,000 L (10,000 gal) in 1952 (Anderson 1990). This volume would amount to approximately 25.4 cm (10 in.) in the dished tank bottom. The tank was filled with unscavenged uranium recovery wastes in 1953. This waste was scavenged and routed to tank 241-C-112 in 1955.

The tank then received ferrocyanide-scavenged waste. The solids from this waste would be high in nickel, calcium, cyanide (as ferrocyanide), ¹³⁷Cs, and uranium, although the uranium may have settled out in the tank originally receiving the UR waste. Because some of the ferrocyanide waste feed was concentrated 1C and CW wastes (EBs), the waste could also be high in aluminum. During this time, several batches of the concentrated wastes that were scavenged for ⁶⁰Co and ⁹⁰Sr were processed without ferrocyanide. However, further processing with ferrocyanide continued until the end of scavenging in early 1958. The estimated solids volume in tank 241-C-109 at the end of the scavenging program was between 216,000 and 341,000 L (57,000 and 90,000 gal). This would amount to 41.1 to 71.6 cm (16.2 to 28.2 in.) distributed evenly across the tank.

The last major waste type was evaporated aluminum cladding waste. While these materials would be high in aluminum and silica, the solids volume of this waste is unknown. The grey/white solids seen in the video recordings of the core extrusions are believed to be cladding waste, and the tan/dark brown solids are thought to be ferrocyanide sludge. Their observed position during extrusion agrees with the historical record. The volume of strontium semiworks waste was small and probably would not have been visually detectable. However, it would have had a relatively high 90 Sr content because it included strontium recovery and purification waste losses. This characteristic would be readily observable in the radiochemistry analyses.

6.2 REVIEW OF THE SUBSEGMENT ANALYTE PROFILES

The following conclusions are drawn from review of the subsegment analyses presented in Section 5.0.

Core 47

Chemical analyses indicate this material is ferrocyanide waste, although the wastes scavenged were mostly evaporated IC (with some CW wastes). The relatively high nickel, calcium, and ¹³⁷Cs levels in the composites and the analyte profiles in the subsegments lead to this conclusion. The extremely high aluminum values are attributed to concentrated IC and coating wastes (both scavenged and unscavenged) deposited in the tank. The phosphate/phosphorus profile indicates very little BiPO, 1C waste in the upper portions of the tank. Phosphate/phosphorus routinely demonstrate an increasing concentration profile as a function of depth. In addition, the total cyanide analysis indicates residual cyanide in the waste although the measured cyanide concentration is much lower than that expected from simulant information. However, the DSC traces show no discernable exotherm in the temperature range where the In Farm simulants begin to show reactions. Instead, the overall energy release is highly endothermic, postulated to be from the decomposition of aluminum hydroxide to alumina and water. High 90Sr values in 47-18 indicate hot semiworks/strontium semiworks and the 90Sr values decrease dramatically as a function of depth.

Core 48

Because the amount of solids recovered from this core was 14.0 cm (5.5 in.) (assumed to be subsegment 1D); thus, the waste origin is somewhat indeterminate. Chemical analyses indicate this material is ferrocyanide waste, the relatively high nickel, calcium, and ¹³⁷Cs levels in the composites and the analyte profiles in the subsegments support this conclusion. However, none of the primary analytes (e.g., nickel, aluminum, calcium, and phosphate), match the other two cores well as composites or the subsegment profiles. The waste from this core has the highest total overall cyanide content and lowest aluminum concentration. It exhibits a minor exotherm in the temperature range where the In Farm simulants begin to show reactions. This material is much more reminiscent of the sample cores from tank 241-C-112 than of the samples from tank 241-C-109. The relatively low ⁹⁰Sr values and the lack of a high value on top of the sample suggest this segment was acquired from deeper in the tank than the other samples.

Core 49

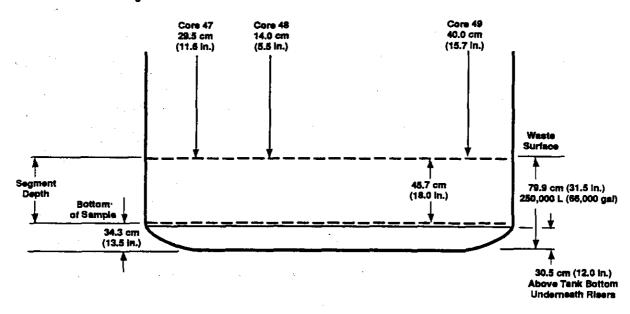
Chemical analyses indicate this material is ferrocyanide waste although the wastes scavenged were mostly evaporated IC (with some CW wastes), much similar to Core 47. The relatively high nickel, calcium, and ¹³⁷Cs levels in the composites and the analyte profiles in the subsegments lead to this conclusion and there is relatively close correspondence between the two cores. The extremely high aluminum values are attributed to concentrated IC and coating wastes (both scavenged and unscavenged) deposited in the tank. The phosphate/phosphorus profile indicates very little IC waste in the upper portions of the tank. Phosphate/phosphorus routinely demonstrate an increasing concentration profile as a function of depth. In addition, the total cyanide analysis indicates residual cyanide in the waste although the measured cyanide concentration is much lower than that expected from simulant information. However, the DSC traces show no discernable exotherm in the temperature range where the In Farm simulants begin to show reactions. Instead, the overall energy release is highly endothermic, postulated to be from the decomposition of aluminum hydroxide to alumina and water. High ⁹⁰Sr values in 49-1B indicate HS/strontium semiworks and the ⁹⁰Sr values decrease dramatically as a function of depth.

6.2.1 Tank Entrance/Exit Effects on Analyte Distribution

Figure 6-1 shows an elevation and a plan of where the core samples were taken. Important items to note are that Cores 47 and 48 were taken from risers near the ferrocyanide waste inlet, while Core 49 was obtained near the waste pumpout riser. The decant "float and flex" pump contained a 6.1-m (20-ft) section of flexible hose that could traverse a relatively wide area on that side of the tank. The cascade fill line (where BiPO, 1C waste entered the tank) is closer to the Core 49 sample point than to the Core 47 and 48 risers. The elevation view shows this is a shallow-dish bottom tank and the bottom of the core samples were 34.3 cm (13.5 in.) above the centerline inside bottom of the tank. Also shown is the waste surface, measured from the centerline as 79.9 ± 1.3 cm $(31.5 \pm 0.5$ in.). Figure 6-2 shows a representation of the overall waste profile of tank 241-C-109 and the assumed volumes, boundaries, and positions of the various individual layers as they are believed to exist.

As new wastes entered the tank and distributed themselves across the tank, the solids under and around the tank pumpout (Core 49) could have been disturbed (and occasionally solids transferred) in behavior similar to the last in-first out principle. However, an inspection of the analyte profiles and model results indicated that disturbance and transfer of ferrocyanide solids did not appear to occur in tank 241-C-109; the waste did not accumulate to levels that the floating suction pump encountered. It is believed that the material beneath the waste inlet (Cores 47 and 48) would have been disturbed initially but over time large stratified layers resistant to mixing would have eventually build up. The Pb and Si rich materials initially settled in the tank, not being as flocculent or as easily suspended as the ferrocyanide solids, may have settled out initially near the inlet. Thus the influence of the waste inlet and outlet locations provides insight to the analyte and waste profiles between Cores 47 and 49.

Figure 6-1. Elevation and Plan of Tank 241-C-109.



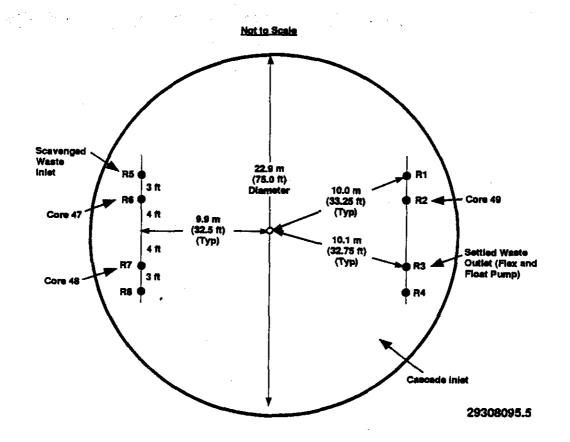
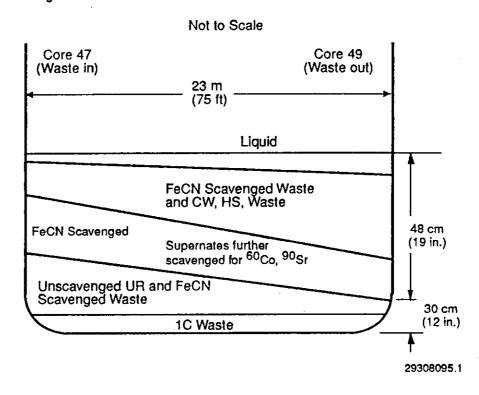


Figure 6-2. Waste Profile of Tank 241-C-109.



- Dished Bottom: First cycle BiPO₄ waste or unscavenged uranium recovery (UR) waste 39,000 L (10,000 gal)
- Tank Layer 1: Scavenged UR and 1C waste 75,000 L (19,800 gal)
- Tank Layer 2: Non-FeCN scavenged, evaporator-processed 1C and CW waste 65,900 L (17,400 gal)
- Tank Layer 3: Evaporator-processed (CW) ferrocyanide scavenged waste and Hot Semiworks waste 56,000 L (14,800 gal)
- Supernatant: 17,000 L (4,500 gal).

The ¹³⁷Cs concentrations vary within a factor of two in the core composites, which is not surprising given the amount of waste that was scavenged without ferrocyanide and its dilutive effect. In addition, the Core 48 waste material appears to be much different in composition than the other two cores, further contributing to the difference. However, the ¹³⁷Cs concentration as a function of depth in Cores 47 and 49 shows profiles consistent with the wastes believed to be associated with the subsegments; low ¹³⁷Cs values for unscavenged wastes (1C and HS), higher ¹³⁷Cs values for ferrocyanide wastes. The ¹³⁷Cs profile shows an increasing trend as a function of depth in both cores. The ⁹⁰Sr concentration for both cores shows an extremely skewed concentration profiles as a function of depth, however, there are no consistently high ⁹⁰Sr values localized around the pumpout riser, as seen in tank 241-C-112,, suggesting that the waste buildup in this tank was not as extensive and the pump did not disturb the waste. The ⁹⁰Sr concentration is extremely high in the top subsegment and then the concentration falls dramatically, which corresponds with the historical fill pattern. The lack of a high ⁹⁰Sr concentration in Core 48 suggests that the sample was obtained from a deeper section of the tank and no surface material was taken.

The upper subsegments of Cores 47 and 49 have extremely high aluminum concentrations. The concentrations seen were initially unexpected, until further investigation revealed that they were evaporator processed 1C and CW (unscavenged UR waste was expected). These concentrations may be typical for evaporator-processed cladding wastes that were deposited on top of the ferrocyanide wastes. Aluminum also shows similar distribution behavior to 90 Sr; a high concentration initially that decreases as a function of depth. It is expected that the bulk of the BiPO4 1C waste lies below the depth that can be core sampled through the available risers. However, the phosphorous and phosphate profiles indicated from the analytical results strongly suggest first decontamination cycle waste is present.

6.3 CALCULATED BULK INVENTORIES OF SELECTED ANALYTES

Several safety issues are defined by certain bulk amounts or weight percent of a given analyte. Tables 6-1 through 6-4 present the calculated bulk amounts of some selected analytes and their weight percent contribution to the waste matrix. The gross waste inventory in the tank is estimated to be 303,000 kg (284,000 kg wet solid, and 19,000 kg of drainable liquid). Appendix A presents the data, assumptions, and calculations used to determine the following values. Estimated volumes, average analyte concentrations, and density measurements for each hypothesized region were used to develop bulk inventory values.

The bulk inventory of disodium nickel ferrocyanide in the wet solids is 6,800 g-mol, assuming the calculated inventory of total cyanide is present as that analyte. Molar ratios for ferrocyanide, nitrate, and nitrite in the wet solids (assuming this value for ferrocyanide) are 1: 27.2: 36.4.

Table 6-1. Energetics Related Analyte Values.

	TOC	Total cyanide	NO ₂	NO ₃	H ₂ O
Bulk inventory (Mg)	0.81	1.12	13.1	13.3	1221
Weight percent (total)	0.27	0.37	4.32	4.38	40.22
Bulk inventory, wet solids (Mg)	0.76	1.10	11.8	11.9	109
Weight percent (wet solids)	0.27	0.39	4.14	4.19	38.22

¹Water content combines interstitial and free water (i.e., supernate).

TOC = Total organic carbon.

Table 6-2. Fission Product Inventory.

	¹³⁷ Cs	⁹⁰ Sr
Bulk inventory (Ci) (wet solids)	221,600	269,900
Heat generation (w)	1,046	1,808

The total heat load of the tank is 2,854 w. The volumetric heat generation rate for the waste in the tank based on the solids volume is 1.2 E-02 w/L.

Table 6-3. Plutonium/Americium Inventory.

	²³⁸ Pu	²³⁹ Pu	²⁴¹ Am
Bulk Inventory (Ci) (wet solids)	0.012	232.9	90.9
Bulk Inventory (g)	7.3 E-04	3,800	26.5

	A1	Ca	Fe	Na	Ni	Р	Рb	Si	U
Bulk inventory (Mg)	19.8	5.5	6.3	23.9	7.6	5.1	1.1	2.2	2.9
Weight percent	7.0	1.9	2.2	8.4	2.7	1.8	0.4	0.8	1.0

Table 6-4. ICP Major Element Inventory (From Fusion Preparation Results).

6.4 COMPARISONS WITH THE BORSHEIM/SIMPSON MODEL ESTIMATES

Calculations of the 137 Cs, nickel, and Fe(CN) $_6^{4-}$ inventories are analytes appropriate for comparison with the model. Assumptions regarding the tank used in the calculations for the analytical estimates, and the calculations themselves, are presented in Appendix A. Table 6-5 presents comparisons of the calculated values with the original and revised Borsheim/Simpson (1991) values after scavenging was finished.

Table 6-5. Comparisons of Initial and Revised Borsheim/Simpson Model Estimates with Values Calculated from Analytical Results.

Analyte	Borsheim/Simpson		Revised Bors	Analytical Estimates	
	Retained	Input	1.0 (retained) vol%	1.5 (retained) vol%	(from Section 6.3)
Ni, moles	30,200	47,300	77,800 ¹	77,800 ¹	131,700 68,900 ²
¹³⁷ Cs, kCi (decayed to 1993)	91.1	142.6	142.6	142.6	221.6
Fe(CN) ₆ , moles	30,200	47,300	47,300	47,300	6,800

Includes the 60Co scavenging contribution.

Several assumptions must be made to calculate the tank contents before making comparisons to the Borsheim/Simpson model predictions for selected analytes. In addition, several assumptions of that model must be examined because they affect the original predictions regarding the waste in the tanks. These assumptions are that (1) 4.25 vol% solids formation occurs (which is representative of the U Plant materials, but found not to be representative of the In Farm waste), (2) no additional settling or compaction; (3) negligible waste transfer (input/output) effects; and (4) transfers after the scavenging program did not meaningfully affect the condition of the waste. However, at

²Based on an average of the ICP acid leach core composites.

the time they were obtained, these data and assumptions were the best available. As the ferrocyanide program evolved, more and better data became available.

The development of the model provided some preliminary understanding to the condition and distribution of the waste in the tank. Generally, the model gave values that were within \pm 50 percent of the values calculated from the analytical results. Where agreement was not good, further investigation found reasonable sources for the difference. The range of values developed from the model was adequate for defining initial conditions and bounding values; however, for analytes like $^{90}\mathrm{Sr}$ and ferrocyanide itself, further process history contributed meaningfully to the present inventory in the tank, as determined from laboratory analysis. While further clarification was provided by physical and chemical characterization of flowsheet materials and aging and energetics studies, in this case the flowsheets are only a general guide to the energetics behavior. There are fundamental differences in the make-up of TBP waste and 1C and CW wastes. The model functioned well within the constraints placed on its operation and it remains flexible enough to run further trials with new parameters, which have been done and are presented in Appendix A.

As noted previously, the analytical nickel values are biased high, perhaps as much as 100 percent, by the use of a nickel crucible in the ICP fusion assay. In addition, ⁶⁰Co scavenging was done in several of the batches that were settled in tank 241-C-109, adding to the nickel inventory but not contributing to the ferrocyanide content. Approximately 30,500 g-mol of additional nickel was added to the tank in these process runs. Therefore, the nickel inventory determined from Borsheim/Simpson (1991) should be adjusted upwards by that amount to account for the additional nickel, because the model only accounted for nickel deposited with ferrocyanide. Agreement between the model values and the analysis-based estimates closes when the analytical bias is considered and with inventory adjustments from the cobalt-scavenging contribution. The ICP acid digestion assay values for the core composites provide concentration values in reasonable agreement with calculated estimates but do not provide a profile of the waste. With all of the caveats associated with it, the nickel assay provides no more than a bounding condition for the ferrocyanide inventory as well as indicating that ferrocyanide was (or is) present.

Values for ¹³⁷Cs from Borsheim/Simpson (1991) only loosely bound the inventories calculated from the analytical results. The calculated inventory can vary somewhat depending on which core's density and concentration values are used in the computation. While no overt biases were found in the analysis, the concentrated nature of some wastes disposed to tank 241-C-109 may have a ¹³⁷Cs concentrations high enough to confound inventory estimates and are biased low.

A large degree of uncertainty is associated with the amount of ferrocyanide waste that may have been disposed to the cribs. The original model run had a large amount of solids being discharged, even though the available records indicate that the discharged effluent had only traces of suspended solids in it. The model basis of 4.25 volume percent solids was responsible for this solids loss, and that percentage has been determined to be flawed for this waste type. Simulant studies indicate that while

4.25 volume percent was an accurate simulation of the U Plant scavenging process, an appropriate solids formation value for the In Farm process is 1.0 to 1.5 volume percent (Jeppson and Wong 1993). This additional information is used to develop better model parameters and waste inventory estimates. A rerun of the model using these new solids formation parameters gives significantly better agreement.

The ferrocyanide inventory calculated from the total cyanide analysis remains 6,800 g-mol. The revised model value for the estimated remaining ferrocyanide of 47,300 g-mol (the estimated total ferrocyanide used in processing waste through tank 241-C-109) is significantly higher than that determined from analytical results. This total cyanide measurement, along with the energetic results, suggests a degradation or aging mechanism of some type.

7.0 QUANTITATIVE/STATISTICAL INTERPRETATION OF THE DATA

7.1 INTRODUCTION

This section contains the results of a statistical analysis of data from three core samples taken from tank 241-C-109. Core 47 consisted of three subsegments (denoted by B, C, and D), core 48 consisted of two subsegments (C and D), and core 49 consisted of three subsegments (B, C, and D). The analytical results from the cores were used to obtain estimates of the mean concentration of analytes in the waste. In addition, the data was used to evaluate the Analytical Chemistry Laboratory's ability to homogenize subsegments and to construct core composite samples. Composite samples for each core were made from homogenized subsegment waste and a single composite sample was made from the drainable liquids. Two measurements, the sample and the duplicate, were taken from each core composite and subsegment aliquot. For the homogenization test, additional samples and duplicates were taken from two different locations within a single aliquot.

To reduce the amount of time and effort necessary to perform a meaningful statistical analysis, a reduced number of analytes of interest were selected. The analytes of interest from the inductively coupled plasma (ICP) analyses are aluminum, calcium, iron, sodium, nickel, lead, uranium, and phosphorous. The ICP acid digestion and water leach analyses were performed on the composite core samples. The ICP potassium hydroxide fusion dissolution analyses were performed on both the subsegments and core composite samples. Radiochemical results for the core composite samples were reported for uranium, ²³⁸Pu, ^{239/240}Pu, ¹³⁷Cs, and ⁹⁰Sr. A radiochemistry analysis on the subsegments was performed only for ¹³⁷Cs and ⁹⁰Sr. Each subsegment and core composite sample was analyzed by ion chromatography (IC) for chloride, nitrite, nitrate, phosphate, and sulfate; total cyanide (CN') was determined by an independent procedure. In the following tables (and in Appendix C), the data are identified by the analysis method and the type of sample preparation (e.g., the notation ICP.acid.Al refers to an ICP analysis, acid digestion for aluminum). The core composite sample results are contained in Table 7-1. The subsegment sample results are contained in Table 7-2. The homogenization test results are contained in Table 7-3. Appendix C contains graphic depictions of the data for core composite and subsegment samples.

A close examination of the data reveal several potential anomalies. The following core composite sample results were an order of magnitude different (lower or higher) than other corresponding core composite sample results.

- Core 48 ICP.acid.Al
- Core 47 ICP.acid.Pb
- Core 48 ICP.fusion.Al
- Core 49 ICP.fusion.Pb
- Core 47 total alpha Pu
- Core 47 Pu-239/240.

Table 7-1. Core Composite Data (Units $\mu g/g$ Except Radionuclides $\mu Ci/g$).

Core		17	4	18	49		
Analysis	1	2	1	2	1	2	
ICP.acid.Al ICP.acid.Ca ICP.acid.Fe ICP.acid.Na ICP.acid.Ni ICP.acid.Pb ICP.acid.U ICP.acid.P	7.41e+04	7.16e+04	6.24e+03	6.60e+03	9.59e+04	7.15e+04	
	1.95e+04	2.05e+04	1.44e+04	1.07e+04	1.38e+04	1.08e+04	
	3.52e+04	2.24e+04	1.39e+04	2.65e+04	8.39e+03	5.90e+03	
	8.15e+04	8.22e+04	8.16e+04	9.35e+04	6.58e+04	9.68e+04	
	1.46e+04	1.49e+04	1.63e+04	1.47e+04	1.31e+04	1.06e+04	
	9.96e+03	7.25e+03	5.86e+02	6.26e+02	9.99e+02	7.28e+02	
	1.05e+04	1.10e+04	1.27e+04	1.74e+04	7.10e+03	5.43e+03	
	1.84e+04	1.84e+04	1.45e+04	1.96e+04	1.17e+04	2.71e+04	
ICP.fusion.Al	1.15e+05	1.19e+05	7.28e+03	9.86e+03	1.20e+05	1.34e+05	
ICP.fusion.Ca	2.44e+04	2.49e+04	1.68e+04	1.85e+04	1.45e+04	1.52e+04	
ICP.fusion.Fe	2.02e+04	2.34e+04	2.38e+04	2.06e+04	9.27e+03	8.94e+03	
ICP.fusion.Na	8.71e+04	8.72e+04	1.07e+05	9.33e+04	8.18e+04	7.13e+04	
ICP.fusion.Pb	7.22e+03	7.34e+03	NA	NA	8.03e+02	8.44e+02	
ICP.fusion.U	8.75e+03	9.61e+03	2.78e+04	2.17e+04	5.59e+03	NA	
ICP.fusion.P	2.02e+04	1.96e+04	2.22e+04	1.82e+04	1.77e+04	1.14e+04	
ICP.water.Al	3.36e+02	4.88e+02	NA	NA	NA	NA	
ICP.water.Ca	1.73e+02	1.94e+02	5.93e+01	5.97e+01	8.92e+01	6.62e+01	
ICP.water.Fe	8.85e+02	8.72e+02	1.13e+03	1.15e+03	8.88e+02	9.44e+02	
ICP.water.Na	6.60e+04	6.96e+04	8.92e+04	7.79e+04	5.89e+04	6.09e+04	
ICP.water.Ni	1.40e+02	1.09e+02	3.34e+01	2.85e+01	5.28e+01	5.28e+01	
ICP.water.P	6.35e+03	7.63e+03	1.19e+04	5.46e+03	4.42e+03	3.90e+03	
Chloride	7.00e+02	7.00e+02	8.00e+02	8.00e+02	7.00e+02	7.00e+02	
Nitrite	3.80e+04	4.00e+04	4.20e+04	4.80e+04	3.80e+04	3.90e+04	
Nitrate	3.70e+04	3.70e+04	4.50e+04	5.10e+04	3.50e+04	3.70e+04	
Phosphate	2.01e+04	2.40e+04	3.59e+04	1.75e+04	1.35e+04	1.20e+04	
Sulfate	7.20e+03	8.10e+02	8.90e+03	9.60e+03	6.20e+03	6.90e+03	
Total cyanide	5.60e+03	5.41e+03	1.41e+04	1.46e+04	5.64e+03	5.59e+03	
U (μg/g) Total alpha (Pu) Sr-90 Pu-238 Pu-239/240 Cs-137/water Cs-137/fusion	1.17e+01	1.22e+01	3.00e+01	2.51e+01	7.63e+00	7.42e+00	
	8.05e-01	9.49e-01	6.95e-02	6.66e-02	6.59e-02	9.21e-02	
	1.05e+03	1.30e+03	1.90e+02	1.90e+02	8.77e+02	9.86e+02	
	4.40e-05	NA	7.15e-06	NA	1.11e-05	NA	
	8.04e-01	9.48e-01	6.95e-02	6.66e-02	6.58e-02	9.20e-02	
	9.07e+00	9.40e+00	7.95e+00	1.07e+01	5.61e+00	4.95e+00	
	8.70e+02	8.77e+02	1.11e+03	9.52e+02	5.47e+02	5.66e+02	

Table 7-2. Subsegment Data (Unites $\mu g/g$ Except Radionuclides $\mu Ci/g$).

Core		47		4	8		49	
Subsegment	В	С	D	С	D	В	С	D
ICP.fusion.Al	1.24e+05	1.20e+05	3.27e+04	7.44e+03	9.60e+03	1.81e+05	9.75e+04	7.35e+04
	1.39e+05	1.21e+05	3.13e+04	7.14e+03	1.01e+04	1.90e+05	9.40e+04	6.82e+04
ICP.fusion.Ca	1.07e+04	1.84e+04	2.88e+04	3.02e+04	1.70e+04	5.40e+03	1.89e+04	2.13e+04
	1.02e+04	1.77e+04	2.72e+04	2.84e+04	1.66e+04	3.21e+03	1.82e+04	2.37e+04
ICP.fusion.Fe	8.25e+04	1.54e+04	1.71e+04	2.28e+04	2.27e+04	1.38e+04	4.33e+03	1.36e+04
	4.42e+04	2.65e+04	1.35e+04	1.72e+04	1.94e+04	1.74e+04	4.82e+03	1.72e+04
ICP.fusion.Na	4.97e+04	6.32e+04	1.02e+05	1.38e+05	1.01e+05	4.51e+04	6.09e+04	9.02e+04
	5.24e+04	6.29e+04	1.04e+05	9.33e+04	1.03e+05	4.10e+04	6.48e+04	9.25e+04
ICP.fusion.Pb	5.53e+03	2.99e+03	1.86e+04	NA	7.24e+02	2.07e+03	NA	6.95e+02
	4.57e+03	2.78e+03	1.00e+04	NA	6.62e+02	1.90e+03	NA	7.62e+02
ICP.fusion.U	1.15e+04	6.61e+03	6.24e+03	1.81e+04	1.46e+04	8.66e+03	NA	1.15e+04
	1.20e+04	5.68e+03	5.44e+03	1.54e+04	1.41e+04	7.15e+03	NA	1.32e+04
ICP.fusion.P	NA	1.26e+04	2.90e+04	2.62e+04	2.02e+04	4.57e+03	1.14e+04	2.05e+04
	7.89e+03	1.23e+04	3.12e+04	2.03e+04	2.16e+04	NA	1.16e+04	2.03e+04
Chloride	5.00e+02	7.00e+02	8.00e+02	1.00e+03	1.00e+03	5.00e+02	8.00e+02	8.00e+02
	6.00e+02	7.00e+02	7.00e+02	9.00e+02	1.00e+03	5.00e+02	8.00e+02	8.00e+02
Nitrite	2.70e+04	3.70e+04	4.00e+04	4.90e+04	4.90e+04	2.58e+04	4.20e+04	4.60e+04
	2.88e+04	3.70e+04	3.90e+04	5.30e+04	5.00e+04	2.71e+04	4.50e+04	4.40e+04
Nitrate	2.69e+04	3.60e+04	3.90e+04	5.50e+04	5.20e+04	2.52e+04	4.00e+04	4.40e+04
	2.83e+04	3.60e+04	3.80e+04	5.70e+04	5.50e+04	2.62e+04	4.40e+04	4.20e+04
Phosphate	7.10e+03	9.60e+03	3.40e+04	1.50e+04	3.80e+04	6.00e+03	8.90e+03	2.43e+04
	7.50e+03	9.50e+03	5.50e+04	1.65e+04	3.40e+04	6.20e+03	8.70e+03	2.60e+04
Sulfate	4.90e+03	7.10e+03	7.60e+03	1.08e+04	1.00e+04	4.50e+03	7.90e+03	7.90e+03
	5.20e+03	7.10e+03	7.10e+03	1.12e+04	1.00e+04	4.80e+03	8.40e+03	8.30e+03
Total cyanide	3.05e+03	4.49e+03	5.83e+03	1.10e+04	8.60e+03	3.50e+03	8.14e+03	5.61e+03
	3.03e+03	4.23e+03	5.82e+03	1.15e+04	8.71e+03	3.57e+03	8.02e+03	5.43e+03
Sr-90.fusion	4.60e+03	4.56e+02	2.31e+02	1.59e+02	1.27e+02	2.56e+03	2.02e+02	1.88e+02
	4.51e+03	4.82e+02	1.99e+02	1.44e+02	1.14e+02	2.23e+03	1.89e+02	1.97e+02
Cs-137.fusion	3.17e+02	8.12e+02	9.71e+02	1.17e+03	1.22e+03	1.21e+02	5.53e+02	6.60e+02
	3.57e+02	7.31e+02	9.23e+02	1.14e+03	1.11e+03	1.15e+02	1.44e+02	7.43e+02

Table 7-3. Homogenization Test Data (Units $\mu g/g$ Except Radionuclides $\mu Ci/g$).

Core	48	icildes µci/g		9
Subsegment	D	D	D	D
Location	1	2	1	2
ICP.acid.Al	8.73e+03	9.45e+03	3.94e+04	4.18e+04
	7.89e+03	8.84e+03	4.66e+04	4.68e+04
ICP.acid.Ca	1.53e+04	1.73e+04	1.46e+04	1.69e+04
	1.42e+04	1.56e+04	1.94e+04	1.87e+04
ICP.acid.Fe	2.38e+04	1.78e+04	8.85e+03	9.05e+03
	1.37e+04	1.68e+04	1.15e+04	1.12e+04
ICP.acid.Na	1.16e+05	9.93e+04	1.16e+05	8.80e+04
	1.21e+05	1.06e+05	8.66e+04	8.01e+04
ICP.acid.Ni	1.71e+04	1.94e+04	1.19e+04	1.29e+04
	1.56e+04	1.74e+04	1.56e+04	1.48e+04
ICP.acid.Pb	6.17e+02	7.23e+02	4.85e+02	5.08e+02
	5.68e+02	6.45e+02	6.70e+02	6.16e+02
ICP.acid.U	1.54e+04	1.74e+04	9.71e+03	1.00e+04
	1.45e+04	1.55e+04	1.34e+04	1.21e+04
ICP.acid.P	2.69e+04	1.96e+04	3.23e+04	2.09e+04
	3.08e+04	2.29e+04	1.86e+04	1.75e+04
ICP.fusion.Al	NA	NA	6.17e+04	5.31e+04
	NA	NA	6.30e+04	5.59e+04
ICP.fusion.Ca	NA	NA	2.17e+04	2.14e+04
	NA	NA	2.22e+04	2.17e+04
ICP.fusion.Fe	NA	NA	1.37e+04	1.28e+04
	NA	NA	1.44e+04	1.28e+04
ICP.fusion.Na	NA	NA	9.08e+04	9.02e+04
	NA	NA	9.05e+04	8.92e+04
ICP.fusion.Ni	NA	NA	N/A	N/A
	NA	NA	N/A	N/A
ICP.fusion.Pb	NA	NA	6.25e+02	6.46e+02
	NA	NA	7.45e+02	6.50e+02
ICP.fusion.U	NA	NA	1.23e+04	1.08e+04
	NA	NA	1.31e+04	1.18e+04
ICP.fusion.P	NA	NA	1.87e+04	1.95e+04
	NA	NA	1.91e+04	1.86e+04
Cs-137.fusion	NA	NA	7.13e+02	6.96e+02
	NA	NA	7.50e+02	7.00e+02
Cs-137.acid	8.52e+00	8.81e+00	3.54e+01	1.93e+01
	1.66e+01	1.43e+01	4.34e+01	2.74e+01

Core 47 subsegment 1B results for ICP.fusion.Fe is at least twice as large as its duplicate and all other subsegment data for this analyte.

The following subsegments were different by an order of magnitude (lower or higher) than other corresponding subsegment results.

- Core 48 subsegments 1C and 1D for ICP.fus.Al
- Core 47 and 48 subsegment 1B for fusion.Sr-90.

There is no direct evidence that the results noted above are because of analytical measurement errors. Consequently, the statistical analysis was performed on the data as it is reported in Tables 7-1, 7-2, and 7-3.

7.2 MEAN CONCENTRATION ESTIMATES

One objective of the characterization effort was to estimate the analyte concentrations in the waste. This task was accomplished by computing the mean concentrations and 95 percent confidence intervals (CIs) on the mean concentrations. The estimated inventory and CI on the inventory of an analyte are the corresponding mean concentration estimates and CI multiplied by the volume of waste in the tank. Bulk inventory estimates based on these values are not given in this document. Table 7-1 contains the core composite data used to compute the mean concentration estimates and the CIs. The NA symbol indicates that the data were not available. Results for ²³⁸Pu were not included in any computations because there were no duplicate measurements.

The concentration estimates are given in the form of 95 percent CIs on the mean concentration. It is assumed that each sample and duplicate is analyzed independently of one another. The two analytical results are used to estimate the analytical measurement error. Because of the hierarchical structure of the data, the analytical measurement error (variance) alone is not the appropriate error term to use in computing the CIs. A linear combination of the analytical measurement variance and spatial variance is the appropriate variance of the mean for the CIs. The variance of the mean is obtained from the analysis of variance (ANOVA) corresponding to the model. The formulas used to calculate these CIs are given in Jensen and Whitcher (1993). Table 7-4 contains the summary statistics, by analyte, for ICP acid digestion, ICP water leach, ICP KOH\Ni fusion dissolution, radiochemistry, and IC analyses.

Table 7-5 contains the summary statistics for the drainable liquid composite sample. The summary statistics are as follows.

- \bar{y} = Arithmetic mean of the concentration data
- $\hat{\sigma}^2(\bar{y}) = \text{Estimated variance of } \bar{y}$
- df = Degrees of freedom associated with BMS
- 95% LL = Lower limit to the 95 percent CI on the mean
- 95% UL = Upper limit to the 95 percent CI on the mean

For some analytes, the lower confidence limit (95% LL) was negative. Because concentrations are strictly greater than or equal to zero, any negative 95 percent LL values were set equal to zero.

The estimated variance of the mean $[\hat{\sigma}^2(\bar{y})]$ is very large relative to the mean for most of the analytes. One cause is the large differences between core composite samples; i.e., the large spatial variability. In Section 7.6, the analytical results from the core composite samples are compared to determine if there are significant differences between cores. A similar comparison is also made between the subsegments within each core.

Table 7-4. Concentration Estimate Statistics (Units $\mu q/q$ Except Radionuclide $\mu Ci/q$).

Analyte	ӯ	∂ ² (ȳ)	df	95% Lower limit	95% Upper limit
ICP.acid.Al ICP.acid.Ca ICP.acid.Fe ICP.acid.Na ICP.acid.Ni ICP.acid.Pb ICP.acid.U	5.43e+04 1.50e+04 1.87e+04 8.36e+04 1.40e+04 3.36e+03 1.07e+04 1.83e+04	5.84e+08 6.38e+06 3.96e+07 4.02e+06 1.23e+06 6.89e+06 6.40e+06 4.73e+05	2 2 2 2 2 2 2 2	0.00e+00 4.10e+03 0.00e+00 7.49e+04 9.27e+03 0.00e+00 0.00e+00	1.58e+05 2.58e+04 4.58e+04 9.22e+04 1.88e+04 1.47e+04 2.16e+04
ICP.fus.Al ICP.fus.Ca ICP.fus.Fe ICP.fus.Na ICP.fus.Pb ICP.fus.U ICP.fus.P	8.40e+04 1.91e+04 1.77e+04 8.79e+04 4.05e+03 1.47e+04 1.82e+04	1.43e+09 8.50e+06 1.85e+07 4.64e+07 1.04e+07 3.72e+07 3.29e+06	2 2 2 2 1 2 2	0.00e+00 6.52e+03 0.00e+00 5.86e+04 0.00e+00 0.00e+00	2.47e+05 3.16e+04 3.62e+04 1.17e+05 4.51e+04 4.09e+04 2.60e+04
ICP.water.Ca	1.07e+02	1.49e+03	2	0.00e+00	2.73e+02
ICP.water.Fe	9.78e+02	6.61e+03	2	6.28e+02	1.33e+03
ICP.water.Na	7.04e+04	4.82e+07	2	4.05e+04	1.00e+05
ICP.water.Ni	6.94e+01	7.98e+02	2	0.00e+00	1.91e+02
ICP.water.P	6.61e+03	1.74e+06	2	9.31e+02	1.23e+04
Chloride	7.33e+02	1.11e+03	2 2 2 2 2 2 2	5.90e+02	8.77e+02
Nitrite	4.08e+04	4.36e+06		3.18e+04	4.98e+04
Nitrate	4.03e+04	1.48e+07		2.38e+04	5.69e+04
Phosphate	2.05e+04	1.68e+07		2.85e+03	3.81e+04
Sulphate	7.70e+03	6.48e+05		4.24e+03	1.12e+04
Total cyanide	8.46e+03	8.39e+06		0.00e+00	2.09e+04
U (µg/g)	1.57e+01	3.69e+01	2 2 2 2 2 2	0.00e+00	4.18e+01
Tot.Alpha.Pu	3.41e-01	7.17e-02		0.00e+00	1.49e+00
Sr-90	7.66e+02	8.77e+04		0.00e+00	2.04e+03
Pu-239/240	3.41e-01	7.16e-02		0.00e+00	1.49e+00
Cs-137.water	7.95e+00	1.78e+00		2.21e+00	1.37e+01
Cs-137.fusion	8.20e+02	1.95e+04		2.20e+02	1.42e+03

		• -		•	
Analyte	ÿ	$\hat{\sigma}^2(\bar{y})$	Degrees of freedom	95% Lower limit	95% Upper limit
ICP.acid.Al ICP.acid.Ca ICP.acid.Fe ICP.acid.Na ICP.acid.Ni ICP.acid.Pb ICP.acid.U ICP.acid.P	1.57e+02 2.09e+02 1.67e+03 9.69e+04 3.44e+02 NA NA 4.20e+03	1.50e+01 1.74e+01 5.56e+02 2.66e+01 1.03e+01 NA NA 9.65e+02	1 1 1 1 1 NA NA	1.08e+02 1.56e+02 1.38e+03 9.69e+04 3.03e+02 0.00 0.00 3.80e+03	2.06e+02 2.62e+02 1.97e+03 9.70e+04 3.84e+02 0.00 0.00 4.59e+03
Chloride Nitrite Nitrate Phosphate Sulphate	1.30e+03 7.10e+04 7.20e+04 1.35e+04 1.28e+04	NA NA NA NA NA	NA NA NA NA	NA NA NA NA	NA NA NA NA NA

Table 7-5. Concentration Estimates Statistics, Drainable Liquid, (Units $\mu g/g$).

7.3 HOMOGENIZATION TEST

Another task in the characterization effort was to evaluate the ability of the Analytical Chemistry Laboratory to homogenize subsegments. Subsegment D, from Cores 47, 48, and 49, was homogenized and arbitrarily divided into two parts. One subsample was obtained from each part. Two aliquots were taken from each subsample and prepared for chemical analysis. The homogenization test data is given in Table 7-3. ICP acid digestion and fusion dissolution analyses were conducted on the samples for the following analytes: aluminum, iron, sodium, nickel, lead, uranium, and phosphorus. Acid digestion and fusion dissolution results were also reported for ¹³⁷Cs.

Because of the nested structure (subsamples within segments, aliquots within subsamples) within the data, a hierarchical statistical model was fit to the data. A description of this type of model is contained in Snedecor and Cochran (1980). Such a model is used to estimate different components of variability in the data. The total variability in the data is decoupled into three components; one because of variability between subsegments, one because of the variability between samples taken from different locations on each homogenized subsegment $[\sigma^2(L)]$, and one because of the analytical measurement error $[\sigma^2(A)]$. The analytical measurement error accounts for the differences between aliquots taken from the same location.

To quantify the contribution of $\sigma^2(L)$ (the component of variability because of location or homogenization), the ANOVA corresponding to the hierarchical model is used. From the ANOVA, a test is constructed to determine if $\sigma^2(L)$ is significantly different from zero. If $\sigma^2(L)$ is significantly different from zero, then the laboratory does not have the ability to homogenize subsegments. If $\sigma^2(L)$ is not significantly different from zero, then the laboratory has the ability to homogenize core segments.

The reason underlying this test is that if $\sigma^2(L)=0$, then the mean concentrations at the two locations are equal, i.e., there is no difference between the locations.

The F-test is used to determine whether or not $\sigma^2(L)$ is significantly different from zero. The p-values (the attained level of significance) from these tests are given in Table 7-6. If the p-value is smaller than 0.05, then $\sigma^2(L)$ is significantly different from zero. In all but one case (ICP.fus.Al), the p-values are greater than 0.05. This indicates that except for this analyte, $\sigma^2(L)$ is not significantly different from zero. Based on the results of this statistical test, it can be concluded that the Analytical Chemistry Laboratory can adequately homogenize core segments. However, it needs to be noted that there is no reference value available to check the degree of homogenization. That is, the differences between the results from the two locations must be within two percent of each other. If such a value were available, the conclusions in this section may not be valid.

	1451	· . · ·	,,,,,,,,						
Test: $\sigma^2(L)=0$ p-value									
Analyte	Aluminum	Calcium	Iron	Sodium	Nickel	Lead	Uranium	Phosphorus	¹³⁷ Cs
Acid	0.890	0.649	0.922	0.229	0.551	0.572	0.667	0.290	0.214
Fusion	0.036	0.389	0.072	0.216	NA	0.606	0.164	0.706	0.092

Table 7-6. Homogenization Test Statistical Results.

7.4 COMPARISON WITH A SIMULATED CORE COMPOSITE MEAN

The ability of the Analytical Chemistry Laboratory to make core composite samples from the individual subsegment samples was also evaluated. Core composite samples were formed by combining aliquots from each homogenized subsegment in the core. Each subsegment is weighted equally in the composite sample. Each core composite sample was homogenized, and a sample and duplicate value were obtained. A simulated core composite (SCC) was statistically constructed to compare to the corresponding core composite sample results. For each analyte and each core, the SCCs are the average of the subsegment results. This mean or average is denoted by $\bar{y}(w)$. The w is used because $\bar{y}(w)$ is generally a weighted mean. However, in this case the weights are all equal.

For each core, the comparison between the core composite and the SCC is made by computing a CI on the difference between the SCC and the mean of the composite sample. If zero is in the CI, then the laboratory can construct core composite samples satisfactorily (i.e., the SCC cannot be statistically distinguished from the core composite sample mean). If zero is not in the CI, then the laboratory cannot satisfactorily construct core composites (i.e., the

two means are significantly different). The CI for this difference is (LL, UL) where the lower limit (LL) and upper limit (UL) values are

$$LL = [\overline{y}(w) - \overline{y}(c)] - t\sqrt{\hat{\sigma}^2[\overline{y}(w) - \overline{y}(c)]}, \quad UL = [\overline{y}(w) - \overline{y}(c)] + t\sqrt{\hat{\sigma}^2[\overline{y}(w) - \overline{y}(c)]}$$

where:

 $\bar{y}(c)$ = Mean of the two core composite sample results t = Percentile point from student's t distribution $\partial^2[\bar{y}(w)-\bar{y}(c)]$ = Is the estimated variance of the difference.

Appendix C outlines the method used to calculate $\hat{\sigma}^2[\bar{y}(w)-\bar{y}(c)]$. The estimated variance $\hat{\sigma}^2[\bar{y}(w)-\bar{y}(c)]$ was calculated using the data from all three cores because of the limited information available. The degrees of freedom (df) associated with t were calculated using Satterthwaite's approximation (Snedecor and Cochran 1980). In the above equations, $\bar{y}(w)$ and $\bar{y}(c)$ should have a subscript indicating the core. To simplify the notation, the subscript was omitted.

Table 7-7 contains summary statistics for all three cores, including the 95 percent CI interval (LL, UL) on the difference between the SCC mean and the core composite mean. All of the CIs on this difference contain zero. This indicates that there is no significant difference between the two means (i.e., the Analytical Chemistry Laboratory can construct a core composite sample from material similar to cores 47, 48, and 49). Note that the CIs on the difference between the two means tends to be rather wide; i.e., LL = -10^5 or -10^4 and UL = $+10^5$ or $+10^4$. The reason for this extreme width is the magnitude of the estimated variance $\partial^2[\bar{y}(w)-\bar{y}(c)]$ and the small number of df. Because the variance is large, the two means would have to be extremely different before zero is not in the CI.

7.5 THE SPATIAL VARIANCE AND ANALYTICAL MEASUREMENT VARIANCE

Using the hierarchical structure of the core composite data, the spatial variance and the analytical measurement variance can be separated from each other. The spatial variance is a measure of the variability between cores. The analytical measurement variance includes among other things, the segment homogenization error, the sample handling error, and the chemical analysis error. This variance is a function of the difference between the analytical results on the sample and duplicate values.

The size of the analytical measurement variance and the spatial variance, along with the df, determines the width of the CIs. The estimate of the variance of the mean is a linear function of the spatial and analytical measurement variances. To help judge the magnitude of these two variance components, this section contains explicit estimates of each variance and CIs for each variance. Estimates of the spatial variance $[\hat{\sigma}^2(S)]$ and analytical measurement variance $[\hat{\sigma}^2(A)]$ were obtained for each analyte using the Restricted Maximum Likelihood Estimation. This method is discussed in further

Table 7-7. Comparison of Simulated Core Composite with the Core Composite.

	e /-/. Compan	i	I	· ·		1	<u> </u>
	A	56.3	5/->	$\hat{\sigma}^2[\bar{y}(w)-$	Degrees	95% Lower	95%
Core	Analyte	ӯ(w)	ÿ(c)	y(c)]	of	limit	Upper
		ļ		3(0)	freedom		limit
47	ICP.fus.Al	9.46e+04	1.17e+05	5.79e+09	3	-2.64e+05	2.20e+05
_ ''	ICP.fus.Ca		2.47e+04		ا ڏ	-2.27e+04	1.10e+04
	ICP.fus.Fe	1	2.18e+04		١ ٥	-1.77e+04	4.05e+04
	ICP.fus.Pe		8.72e+04		3	-6.27e+04	3.29e+04
			7.28e+03		'2	-2.34e+04	2.37e+04
	ICP.fus.Pb				2		4.50e+04
	ICP.fus.U		9.04e+03		6 8 7 2 2 9 5 7 5	-4.72e+04	1
	ICP.fus.P		1.99e+04		9	-1.63e+04	1.02e+04
	Chloride		7.00e+02		5	-3.48e+02	2.81e+02
	Nitrite		3.90e+04			~1.99e+04	1.15e+04
	Nitrate		3.70e+04		<u>5</u>	-2.75e+04	
	Phosphate		2.21e+04		7	-2.71e+04	
	Sulfate		7.30e+03		5	-5.81e+03	4.21e+03
	Total cyanide	4.41e+03	5.51e+03	2.89e+07	2	-2.42e+04	2.20e+04
	Sr-90		1.18e+03	1.09e+06	5 2 9 5	-1.79e+03	2.93e+03
	Cs-137.fus		8.74e+02		5	-1.07e+03	6.98e+02
48	ICP.fus.Al	8.56e+03			4	-2.24e+05	2.24e+05
,	ICP.fus.Ca			5.84e+07	7 8	-1.27e+04	
	ICP.fus.Fe			2.12e+08	8	-3.52e+04	3.18e+04
	ICP.fus.Na	1.09e+05			7	-4.62e+04	6.39e+04
l	ICP.fus.Pb	6.93e+02	NA	4.83e+07	NA	NA	NA
	ICP.fus.U	1.55e+04			2	-5.60e+04	3.76e+04
	ICP.fus.P	2.21e+04	2.02e+04	4.63e+07	9	-1.35e+04	1.73e+04
	Chloride	9.75e+02	8.00e+02	2.08e+04	5	-1.96e+02	5.46e+02
	Nitrite	5.03e+04	4.50e+04	5.95e+07	6	-1.36e+04	2.41e+04
	Nitrate			1.15e+08	5	-2.08e+04	3.43e+04
- 1	Phosphate		2.67e+04		8	-2.90e+04	2.73e+04
- 1	Sulfate		9.25e+03		5	-4.34e+03	6.84e+03
	Total cyanide				3	-2.20e+04	1.32e+04
i	Sr-90	1.36e+02			٥	-2.82e+03	2.72e+03
.	Sr-90 Cs-137.fus		1.03e+02		2956585395	-8.64e+02	1.12e+03
49	ICP.fus.Al	1.17e+05	1.27e+05	5.79e+09	3	-2.51e+05	2.33e+05
	ICP.fus.Ca	1.51e+04	1.49e+04	4.75e+07	6	-1.66e+04	
i	ICP.fus.Fe	1.19e+04		1.60e+08	8	-2.64e+04	3.19e+04
1	ICP.fus.Na	6.58e+04		4.08e+08	7	-5.85e+04	3.70e+04
	ICP.fus.Pb	1.36e+03		3.45e+07		-1.82e+04	1.92e+04
	ICP.fus.U	1.24e+04		1.18e+08) 2	-4.00e+04	5.36e+04
	ICP.fus.P	1.21e+04		3.41e+07	3 2 9 5 7 5 7	-1.57e+04	1.08e+04
	Chloride	7.00e+02		1.50e+04	 	-3.14e+02	3.14e+02
					3		
	Nitrite	3.83e+04			'=	-1.59e+04	1.55e+04
	Nitrate	3.69e+04		9.11e+07	2	-2.36e+04	2.54e+04
	Phosphate		1.28e+04	1.16e+08	/	-2.49e+04	2.61e+04
,	Sulfate		6.55e+03	3.80e+06	5 2	-4.59e+03	5.43e+03
	Total cyanide		5.62e+03	2.89e+07	2	-2.30e+04	2.32e+04
	Sr-90	9.28e+02	9.32e+02	1.09e+06	9	-2.36e+03	2.36e+03
]	Cs-137.fus	3.89e+02	5.57e+02	1.19e+05	5	-1.05e+03	7.19e+02

df = Degrees of freedom.

detail in Harville (1977). In addition, general methods have been outlined that can be used to obtain CIs for $\hat{\sigma}^2(S)$ and $\hat{\sigma}^2(A)$ (Snedecor and Cochran 1980). These CI techniques are the methods used in this document. The CI for $\hat{\sigma}^2(S)$ is approximate, however the CI for $\hat{\sigma}^2(A)$ is exact.

Tables 7-8 and 7-9 contain estimates of the variance components and their 95 percent CIs. For 85 percent of the analytes (28 out of 33), the estimates of spatial variance are larger than those for the analytical error. This large spatial variability contributes to the extreme width of the CI for the mean concentrations and the CI on the difference between the mathematically-derived core composite and the actual core composite sample.

7.6 MULTIPLE COMPARISONS: CORE COMPOSITE SAMPLES AND SUBSEGMENT SAMPLES

A group of statistical methods known as multiple comparisons can be used to determine whether or not there are significant differences between core composite samples and between subsegment samples. These differences will help determine the heterogeneity or layers within the waste. In addition, if significant differences exist between the core composite samples or the subsegment samples, this will help explain the extreme width of the CIs; i.e., it will help explain the large spatial variability. The multiple comparison procedure known as Tukey's Honestly Significant Difference (HSD) (Petersen 1985) was used. The HSD procedure determines if there are significant differences between core composite samples and between subsegment samples. The core composite samples and subsegment samples that are not significantly different from each other can then be grouped together.

For each analyte, HSD comparisons were made between the means of the core composite samples. These comparisons, along with the means for each core composite sample, are contained in Table 7-10. The symbols α and β are used to indicate groupings. The means of core composite samples with the same symbol cannot be statistically distinguished from each other. Core composite samples with different symbols are significantly different from each other. For a given analyte, the core composite samples with an α have a smaller mean concentration that the core composite samples with a β . A dash indicates that no data were available. The HSD comparisons are not based on the spatial variance. They are however, a function of the analytical measurement variance.

From Table 7-10, it is evident that the mean concentration of 12 out of 31 (39 percent) analytes are not significantly different between the cores. That is, for these analytes there is no evidence of heterogeneity within the waste. For the remaining 19 (61 percent) analytes there is a significant difference between the mean concentrations. This indicates significant heterogeneity or spatial variability within the waste. These significant differences inflate the between core variance (spatial variance). This inflated variance helps explain why the CIs are so wide.

Table 7-8. 95 Percent Confidence Interval on $\sigma^2(A)$, Analytical Error Variance.

Analyte	σ̂ ² (A)	degrees of freedom	95% Lower limit	95% Upper limit
ICP.acid.Al ICP.acid.Ca ICP.acid.Fe ICP.acid.Na ICP.acid.Ni ICP.acid.Pb ICP.acid.U	1.00e+08 3.91e+06 5.45e+07 1.20e+08 1.47e+06 1.23e+06 4.18e+06 2.75e+07	333333333	3.21e+07 1.25e+06 1.75e+07 5.89e+07 4.72e+05 3.96e+05 1.34e+06 1.41e+07	1.39e+09 5.43e+07 7.57e+08 2.55e+09 2.04e+07 1.71e+07 5.81e+07 6.09e+08
ICP.fus.Al ICP.fus.Ca ICP.fus.Fe ICP.fus.Na ICP.fus.Pb ICP.fus.U ICP.fus.P	3.71e+07 6.47e+05 3.38e+06 4.97e+07 3.71e+03 9.45e+06 9.43e+06	3 3 3 2 2 2 3	1.19e+07 2.08e+05 1.08e+06 1.59e+07 1.00e+03 2.57e+06 3.03e+06	5.15e+08 8.98e+06 4.70e+07 6.90e+08 1.47e+05 3.74e+08 1.31e+08
ICP.water.Ca ICP.water.Fe ICP.water.Na ICP.water.Ni ICP.water.P	1.67e+02 6.20e+02 2.40e+07 1.66e+02 7.27e+06	3 3 3 3	5.35e+01 1.99e+02 7.70e+06 5.33e+01 2.33e+06	2.32e+03 8.62e+03 3.33e+08 2.31e+03 1.01e+08
Chloride Nitrite Nitrate Phosphate Sulphate Total cyanide	0.00 6.83e+06 6.67e+06 5.93e+07 1.70e+05 2.14e+04	3 3 3 3 3	0.00 2.19e+06 2.14e+06 1.90e+07 5.45e+04 6.88e+03	0.00 9.49e+07 9.26e+07 8.24e+08 2.36e+06 2.98e+05
U (μg/g) Tot.Alpha.Pu Sr-90 Pu-239/240 Cs-137.water Cs-137.fusion	4.05e+00 3.57e-03 1.24e+04 3.57e-03 1.35e+00 4.23e+03	333333	1.30e+00 1.15e-03 3.98e+03 1.15e-03 4.34e-01 1.36e+03	5.63e+01 4.96e-02 1.72e+05 4.96e-02 1.88e+01 5.87e+04

Table 7-9. 95 Percent Confidence Interval on $\sigma^2(S)$, Spatial Variance.

Analyte	ô²(S)	degrees of freedom	p-value	95% Lower limit	95% Upper limit
ICP.acid.Al ICP.acid.Ca ICP.acid.Fe ICP.acid.Na ICP.acid.Ni ICP.acid.Pb ICP.acid.U	1.70e+09	2	0.008	2.57e+08	6.91e+10
	1.72e+07	2	0.048	0.00	7.54e+08
	9.14e+07	2	0.130	0.00	4.66e+09
	0.00	2	0.882	0.00	3.84e+08
	2.95e+06	2	0.110	0.00	1.45e+08
	2.00e+07	2	0.009	2.92e+06	8.16e+08
	1.71e+07	2	0.053	0.00	7.57e+08
	0.00	2	0.939	0.00	3.40e+07
ICP.fus.Al ICP.fus.Ca ICP.fus.Fe ICP.fus.Na ICP.fus.Pb ICP.fus.U ICP.fus.P	4.27e+09 2.52e+07 5.39e+07 1.14e+08 2.08e+07 9.80e+07 5.16e+06	2 2 2 2 1 2 2	0.001 0.003 0.009 0.097 0.000 0.052 0.270	1.08e+09 5.50e+06 7.73e+06 0.00 4.13e+06 0.00	1.69e+11 1.01e+09 2.19e+09 5.48e+09 2.13e+10 4.26e+09 3.85e+08
ICP.water.Ca	4.40e+03	2	0.004	8.52e+02	1.77e+05
ICP.water.Fe	1.95e+04	2	0.003	4.03e+03	7.83e+05
ICP.water.Na	1.32e+08	2	0.037	0.00	5.70e+09
ICP.water.Ni	2.31e+03	2	0.011	2.89e+02	9.45e+04
ICP.water.P	1.60e+06	2	0.364	0.00	2.03e+08
Chloride	3.33e+03	2	0.000	9.03e+02	1.32e+05
Nitrite	9.67e+06	2	0.149	0.00	5.13e+08
Nitrate	4.10e+07	2	0.032	0.00	1.75e+09
Phosphate	2.08e+07	2	0.321	0.00	1.96e+09
Sulphate	1.86e+06	2	0.015	1.58e+05	7.66e+07
Total cyanide	2.52e+07	2	0.000	6.78e+06	9.94e+08
U (µg/g)	1:09e+02	2	0.004	2.12e+01	4.37e+03
Tot.Alpha.Pu	2.13e-01	2	0.001	5.06e-02	8.50e+00
Sr-90	2.57e+05	2	0.006	4.45e+04	1.04e+07
Pu-239/240	2.13e-01	2	0.001	5.04e-02	8.48e+00
Cs-137.water	4.66e+00	2	0.064	0.00	2.10e+02
Cs-137 fusion	5.63e+04	2	0.012	6.66e+03	2.30e+06

Table 7-10. Tukey's HSD Multiple Comparisons, Core Composite Data (Units $\mu g/g$ Except Radionuclides $\mu Ci/g$).

		Group	,	C Rad Tollac I			
Core	47	48	49	47	48	49	RSD
ICP.acid.Al ICP.acid.Ca ICP.acid.Fe ICP.acid.Na ICP.acid.Ni ICP.acid.Pb ICP.acid.U ICP.acid.P	β α α α α α	α α α α β β	β α α α Β α	7.3e+04 2.0e+04 2.9e+04 8.2e+04 1.5e+04 8.6e+03 1.1e+04 1.8e+04	6.4e+03 1.3e+04 2.0e+04 8.8e+04 1.6e+04 6.1e+02 1.5e+04 1.7e+04	8.4e+04 1.2e+04 7.1e+03 8.1e+04 1.2e+04 8.6e+02 6.3e+03 1.9e+04	18% 13% 39% 16% 9% 33% 19% 36%
ICP.fus.Al ICP.fus.Ca ICP.fus.Fe ICP.fus.Na ICP.fus.Pb ICP.fus.U ICP.fus.P	8 8 8 α 8 α	α β α - α	β α α α α	1.2e+05 2.5e+04 2.2e+04 8.7e+04 7.3e+03 9.2e+03 2.0e+04	8.6e+03 1.8e+04 2.2e+04 1.0e+05 NA 2.5e+04 2.0e+04	1.3e+05 1.5e+04 9.1e+03 7.7e+04 8.2e+02 2.8e+03 1.5e+04	7% 4% 10% 8% 1% 28% 17%
ICP.water.Ca ICP.water.Fe ICP.water.Na ICP.water.Ni ICP.water.P	Β α αΒ Β α	α Β Β α	α α α α	1.8e+02 8.8e+02 6.8e+04 1.2e+02 7.0e+03	6.0e+01 1.1e+03 8.4e+04 3.1e+01 8.7e+03	7.8e+01 9.2e+02 6.0e+04 5.3e+01 4.2e+03	12% 3% 7% 19% 41%
Chloride Nitrite Nitrate Phosphate Sulphate Total cyanide	α α α α α	8 & B & B	α α α α α	7.0e+02 3.9e+04 3.7e+04 2.2e+04 7.3e+03 2.8e+03	8.0e+02 4.5e+04 4.8e+04 2.7e+04 9.3e+03 2.1e+02	7.0e+02 3.9e+04 3.6e+04 1.3e+04 6.6e+03 2.8e+03	0% 6% 6% 38% 5% 167%
U(μg/g) Tot.Alpha.Pu Sr-90 Cs-137.water Cs-137.fusion	α Β Β α Β	გ ~ ~ 8	α α β α	1.2e+01 8.8e-01 1.2e+03 9.2e+00 8.7e+02	2.8e+01 6.8e-02 1.9e+02 9.3e+00 1.0e+03	7.5e+00 7.9e-02 9.3e+02 5.3e+00 5.6e+02	13% 18% 15% 15% 8%

RSD = Relative standard deviation.

Examples of the interpretation of the HSD comparisons are as follows. In Table 7-10, aluminum (ICP.acid) has an α for core 48 and a β for core 47 and 49. That is, the mean concentration of aluminum in core 48 is significantly different from the mean concentration in core 47 and 49. The mean concentration of aluminum in core 47 and 49 cannot be distinguished from each other. Another example is uranium (ICP.acid). In Table 7-10, core 48 has a β , core 49 has an α , and core 47 has an $\alpha\beta$. That is, the mean concentration of uranium for core 48 is significantly different from the mean concentration of uranium is not significantly different from the mean concentration in core 48 and it is not significantly different from the mean concentration in core 49.

There were three subsegment samples from cores 47 and 49 (denoted by B, C, and D) and two from core 48 (denoted by C and D). The relative location of the subsegments are given in the Table 7-11.

Table 7-11. Subsegment Assignment for Tukey's HSD.

Core	47	48	49
Subsegment	B C D	C D	B C D

Tukey's HSD procedure was also used to make comparisons between the individual subsegment means. These comparisons, along with subsegment means, are given in Table 7-12. For a given analyte, the relative locations of the subsegments have the form given in the above table. The symbols used to denote groupings of means concentrations are α , β , γ , δ , ϵ , and ϕ . A dash indicates that no data were available. The interpretation and ranking of the groupings are identical to that given for Table 7-10.

The results given in Table 7-12 appear to be complicated. A partial explanation is as follows. The multiple comparisons indicate that the three subsegments between cores 47 and 49 match for sodium, lead, chloride, nitrate, phosphate, and ¹³⁷Cs. Subsegments B and D match between cores 47 and 49 for iron, nitrite, sulfate, and total cyanide. Subsegments C and D match between cores 47, 48, and 49 for iron and ⁹⁰Sr. This is some evidence of layers in the waste. The multiple comparisons for the other analytes indicate waste heterogeneity. As previously stated, such differences inflate the spatial variance. There may be patterns in Table 7-12, other than those indicated, showing layers within the waste.

7.7 COMPARISON OF RESULTS: SINGLE-SHELL TANKS 241-C-109 AND 241-C-112

This section contains a comparison of summary statistics computed from core composite data from tanks 241-C-109 and 241-C-112. Comparisons are made on the mean concentration, the analytical measurement variance, and the spatial variance. Comparisons are also presented for the relative standard deviations and the two variances as a percent of the total variance. Appendix C contains graphs of the data from tank 241-C-109 and 241-C-112. Each core from tank 241-C-109 is paired, according to location within the tank, with a core from tank 241-C-112. Table 7-13 shows this pairing.

Table 7-12. Tukey's HSD Multiple Comparisons, Subsegment Data (Units $\mu g/g$ Except Radionuclides $\mu Ci/g$).

	(Units $\mu g/g$ Except Radionuclides $\mu Ci/g$).						
Core		Group			Mean		RSD
	47	48	49	47	48	49	- KSD
ICP.fus.Al	€		φ	1.3e+05		1.9e+05	6%
	ϵ	α	δ	1.2e+05	7.3e+03	9.6e+04	
	В	α	Y	3.2e+04	9.8e+03	7.1e+04	1
ICP.fus.Ca	ß		α	1.0e+04		4.3e+03	6%
	(Y	ϵ	γδ	1.8e+04	2.9e+04	1.9e+04	[
	€	Y	δ	2.8e+04	1.7e+04	2.3e+04	
ICP.fus.Fe	ß	ļ	α	6.3e+04		1.6e+04	46%
1	α	α	α	2.1e+04	2.0e+04	4.6e+03	
	α	α	α	1.5e+04	2.1e+04	1.5e+04	
ICP.fus.Na	αβ		α	5.1e+04		4.3e+04	14%
1	αβγ	δ	αβy	6.3e+04	1.2e+05	6.3e+04	
	γδ	γδ	βyδ	1.0e+05	1.0e+05	9.1e+04	1
ICP.fus.Pb	α		α	5.0e+03		2.0e+03	50%
	α	} -	-	2.9e+03	NA	l na	
	α	α	α	1.4e+04	6.9e+02	7.3e+02	1
ICP.fus.U	γδ		αβγ	1.2e+04		7.9e+03	9%
	αß	ϵ	[-	6.le+03	1.7e+04	l na	
	α	δε	γδ_	5.8e+03	1.4e+04	1.2e+04	1
ICP.fus.P			_	NA		NA	9%
	αß	Y	α	1.2e+04	2.3e+04	1.1e+04	
	δ	Y	βy	3.0e+04	2.1e+04	2.0e+04	
Chloride	αβ	}	α	5.5e+02		5.0e+02	6%
ļ	βγ	$\delta\epsilon$	γδ	7.0e+02	9.5e+02	8.0e+02	
	γ	€	γδ	7.5e+02	1.0e+03	8.0e+02	
Nitrite	α		α	2.8e+04		2.6e+04	4%
}	В	€	γ	3.7e+04	5.le+04	4.4e+04	}
	Βy	$\delta\epsilon$	γδ	4.0e+04	5.0e+04	4.5e+04)
Nitrate	α		α	2.8e+04		2.6e+04	4%
ĺ	ß	δ	By	3.6e+04	5.6e+04	4.2e+04	[[
	yß	δ	γ	3.9e+04	5.4e+04	4.3e+04	{
Phosphate	α]	α	7.3e+03		6.le+03	28%
	α	αß	α	9.6e+03	1.6e+04	8.8e+03	
	Y	By	αβγ	4.5e+04	3.6e+04	2.5e+04	1 1
Sulfate	α		α	5.le+03		4.7e+03	3%
	ß	$ \epsilon $	Y	7.1e+03	1.le+04	8.2e+03]
·	By	δ	γ	7.4e+03	1.0e+04	8.1e+03	
Total cyanide	α		α	3.2e+03		3.5e+03	3%
	ß	ϵ	δ	4.4e+03	1.1e+04	8.le+03	
	_ Y	δ	γ	5.8e+03	8.7e+03	5.5e+03	
Sr-90	δ		γ	4.6e+03		2.4e+03	8%
	В	αВ	αβ	4.7e+02	1.5e+02	2.0e+02	-//
<u></u>	αβ	α	αβ	2.2e+02	1.2e+02	1.9e+02	
Cs-137.fusion	αВ		α	3.4e+02		1.2e+02	16%
	βγδ	δ	αß	7.7e+02	1.2e+03	3.5e+02	/ /
	γδ	δ	βγ	9.5e+02	1.2e+03	7.0e+02	1
000 0 1							

RSD = Relative standard deviation.

Table 7-13. Core Composite Assignment for Tank Comparison.

Tank	Pair 1	Pair 2	Pair 3
241-C-109	Core 49	Core 48	Core 47
241-C-112	Core 34	Core 35	Core 36

To help compare the two sets of data, paired cores have the same symbols.

7.7.1 Comparison of Mean Concentrations

For the analytes of interest in tank 241-C-109, the mean concentration (\bar{y}) and the variance of the mean concentration $[\hat{\sigma}^2(\bar{y})]$ are presented in Table 7-4. These summary statistics are based on the core composite data. The corresponding results for tank 241-C-112 are contained in Simpson, Borsheim, and Jensen (1993). To test the equality of the mean concentration of the analytes in the two tanks, a 95 percent CI was computed on the difference between the two mean concentrations. The test of equality of mean concentrations is if zero is in the CI, then the two means cannot be distinguished from each other at the 0.05 level of significance. If zero is not in the CI the two means are significantly different from each other.

A 95 percent CI on the difference between the mean concentrations was computed for 29 analytes. The CI was based on Cochran's approximation to the Behrens-Fisher problem (Snedecor and Cochran 1980). Zero was in all of the intervals. Because of the magnitude of the variances of the difference, $\partial^2[\bar{y}(\text{Cl09})-\bar{y}(\text{Cl12})]=\partial^2[\bar{y}(\text{Cl09})]+\partial^2[\bar{y}(\text{Cl12})]$ and the small number of df, the width of the CIs was very large. The lower limits of the intervals tended to be -10^5 or -10^4 and the upper limits tended to be $+10^5$ or $+10^4$. These intervals are a function of both the analytical measurement variance and the spatial variance.

To reduce the magnitude of the width of the CIs, they were also computed using only the analytical measurement variance. The new lower limits of the intervals were reduced to -10^4 or -10^3 and the upper limits were reduced to $+10^4$ or $+10^3$. Zero was in all but two of the new CIs. The two intervals that did not contain zero were for aluminum (ICP.fusion) and $^{239/240}$ Pu. Tables containing the CIs on the difference are not given in this document.

The interpretation of these results is that except for possibly aluminum (ICP.fusion) and ^{239/240}Pu, the mean concentration of the analytes in the two tanks cannot be distinguished from each other. It needs to be emphasized that these comparisons are based on very large spatial and analytical measurement variances and very small df. Consequently, such comparisons may not be meaningful.

7.7.2 Comparison of Variances

The estimates of the analytical measurement variances $[\hat{\sigma}^2(A)]$ for both tank 241-C-109 and 241-C-112 are given in Table 7-14. A special form of the F-test (Snedecor and Cochran 1980) was used to test the equality of these two

Table 7-14. Comparison of Analytical Error Variances, Tanks 241-C-109 and 241-C-112.

Tank	C-109		C-112		
Analyte	ô²(A)	df	∂²(A)	df	p-value
ICP.acid.Al ICP.acid.Ca ICP.acid.Fe ICP.acid.Na ICP.acid.Ni ICP.acid.Pb ICP.acid.U	1.00e+08 3.91e+06 5.45e+07 1.20e+08 1.47e+06 1.23e+06 4.18e+06 2.75e+07	3333333	3.10e+07 1.44e+06 2.76e+07 2.39e+07 1.06e+06 2.41e+04 1.10e+08 3.83e+06	~~~~	0.180 0.217 0.294 0.294 0.399 0.004 0.012
ICP.fus.Al ICP.fus.Ca ICP.fus.Fe ICP.fus.Na ICP.fus.Pb ICP.fus.U ICP.fus.P	3.71e+07 6.47e+05 3.38e+06 4.97e+07 3.71e+03 9.45e+06 9.43e+06	3 3 3 2 2 3	5.67e+05 1.56e+05 9.81e+06 3.96e+06 1.47e+05 1.64e+07	2 2 2 2 2 2 2 2	0.015 0.200 0.192 0.075 0.025 0.366 0.014
ICP.water.Al ICP.water.Ca ICP.water.Fe ICP.water.Na ICP.water.Ni ICP.water.U ICP.water.P	NA 1.67e+02 6.20e+02 2.40e+07 1.66e+02 NA 7.27e+06	NA 3 3 3 3 NA 3	3.05e+04 3.78e+04 3.43e+03 5.11e+08 1.29e+03 2.60e+06 1.13e+07	2 2 2 2 2 2 2	NA 0.000 0.099 0.017 0.065 NA 0.345
Chloride Nitrite Nitrate Phosphate Sulphate Total cyanide	NA 6.83e+06 6.67e+06 5.93e+07 1.70e+05 2.14e+04	NA 3 3 3 3	2.50e+04 1.33e+08 2.41e+08 1.10e+08 6.25e+06 NA	2 2 2 2 2 NA	NA 0.019 0.008 0.298 0.008 NA
U (μg/g) Tot.Alpha.Pu Sr-90 Pu-238 Pu-239/240 Cs-137.water Cs-137.fusion	4.05e+00 3.57e-03 1.24e+04 NA 3.57e-03 1.35e+00 4.23e+03	3 3 3 NA 3 3	5.30e+07 NA 1.36e+03 1.60e-05 2.69e-05 NA 2.31e+02	2 NA 2 2 2 NA 2	0.000 NA 0.101 NA 0.007 NA 0.001

variances. The p-value, which is the attained level of significance of the F-test, is also given in Table 7-14. If the attained level of significance is less than 0.025 (in this special test), then the two analytical measurement variances are significantly different from each other at the 0.05 level of significance. In 13 out of 28 tests (46 percent) the analytical measurement variances were significantly different from each other for the two tanks.

The equality of the spatial variances for the two tanks was tested in a similar manner. Table 7-15 contains the estimates of the spatial variances $[\hat{\sigma}^2(S)]$ and the p-values for the F-test. The spatial variances were significantly different from each other in only four out of 25 tests (16 percent). These variances were significantly different for uranium (ICP.acid), for nickel (ICP.water) and for uranium and $^{239/240}$ Pu. The spatial variances cannot be distinguished from each other for the other analytes.

The implication of these results are that, at least for these two tanks, the degree of heterogeneity in the waste is very similar. The analytical measurement error is not consistent between the two tanks, even though the data were analyzed by the same laboratory. These results must be interpreted with caution, because there are only two df associated with each spatial variance and two or three df for each analytical measurement variance. The df are very small.

Results given in Tables 7-16 and 7-17 may help in the interpretation of the relative magnitude of the estimates of the analytical and spatial variances. For both tanks 241-C-109 and 241-C-112, Table 7-16 gives $\hat{\sigma}^2(A)$ and $\hat{\sigma}^2(S)$ as a percent of the total variance associated with any observation. That is, $\hat{\kappa}\hat{\sigma}^2(A)=100\hat{\sigma}^2(A)/[\hat{\sigma}^2(A)+\hat{\sigma}^2(s)]$ and $\hat{\kappa}\hat{\sigma}^2(S)=100\hat{\sigma}^2(S)/[\hat{\sigma}^2(A)+\hat{\sigma}^2(s)]$. Generally, $\hat{\sigma}^2(s)$ represents the greatest percentage of the total variability for both tanks 241-C-112 and 241-C-109. The relative standard deviations (RSDs) for both tanks are given in Table 7-17. An RSD is a standard deviation expressed as a percent of the mean concentration. That is, RSD(\bar{y})=100* $\hat{\sigma}(\bar{y})/\bar{y}$, RSD(A)=100* $\hat{\sigma}(A)/\bar{y}$, and RSD(S)=100* $\hat{\sigma}(S)/\bar{y}$. The RSD(A) appears to be relatively consistent between the two tanks. The RSD(S) appears to be variable with no apparent pattern.

7.8 SUMMARY OF STATISTICAL INTERPRETATION

For 16 out of 17 analytes, the variability between sampling locations could not be distinguished from zero. Based on the results of this statistical test, it is concluded the Analytical Chemistry Laboratory can satisfactorily homogenize core segments.

Based on the large spatial variance and analytical measurement variance, the comparisons showed that the Analytical Chemistry Laboratory's ability to construct core composite samples was satisfactory.

In 28 out of 33 cases (85 percent) the spatial variability in the core composite samples was larger than the analytical error.

Table 7-15. Comparison of Spatial Variances, Tanks 241-C-109 and 241-C-112.

Tank	C-109		C-112		
Analyte	ĝ²(S)	Degrees of freedom	ô²(S)	Degrees of freedom	p-value
ICP.acid.Al ICP.acid.Ca ICP.acid.Fe ICP.acid.Na ICP.acid.Ni ICP.acid.Pb ICP.acid.U ICP.acid.P	1.70e+09 1.72e+07 9.14e+07 0.00 2.95e+06 2.00e+07 1.71e+07 0.00	2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	3.67e+08 2.49e+07 0.00 2.66e+08 1.55e+07 2.28e+06 1.56e+09 4.52e+07	2 2 2 2 2 2 2 2	0.178 0.408 NA NA 0.160 0.102 0.011 NA
ICP.fus.Al ICP.fus.Ca ICP.fus.Fe ICP.fus.Na ICP.fus.Pb ICP.fus.U ICP.fus.P	4.27e+09 2.52e+07 5.39e+07 1.14e+08 2.08e+07 9.80e+07 5.16e+06	2 2 2 2 1 2 2	4.70e+08 4.09e+07 1.65e+07 5.24e+08 3.98e+06 1.78e+09 8.04e+07	2 2 2 2 2 2 2	0.099 0.382 0.234 0.179 0.149 0.052 0.060
ICP.water.A1 ICP.water.Ca ICP.water.Fe ICP.water.Na ICP.water.Ni ICP.water.U ICP.water.P	NA 4.40e+03 1.95e+04 1.32e+08 2.31e+03 NA 1.60e+06	NA 2 2 2 2 2 NA 2	7.06e+04 0.00 2.26e+05 5.01e+08 8.56e+04 4.55e+06 3.50e+07	2 2 2 2 2 2 2	NA NA 0.079 0.209 0.026 NA 0.044
Chloride Nitrite Nitrate Phosphate Sulphate Total cyanide	3.33e+03 9.67e+06 4.10e+07 2.08e+07 1.86e+06 2.52e+07	2 2 2 2 2 2 2	5.25e+04 1.04e+08 2.21e+08 2.97e+08 9.17e+06 NA	2 2 2 2 2 NA	0.060 0.085 0.156 0.065 0.168 NA
U (μg/g) Tot.Alpha.Pu Sr-90 Pu-238 Pu-239/240 Cs-137.water Cs-137.fusion	1.09e+02 2.13e-01 2.57e+05 NA 2.13e-01 4.66e+00 5.63e+04	2 2 2 NA 2 2 2	2.88e+09 NA 2.96e+06 7.81e-03 4.15e-03 NA 2.47e+03	1 NA 2 2 2 2 NA 2	0.000 NA 0.080 NA 0.019 NA 0.042

Table 7-16. Comparison of Percent Variance, 241-C-109 and 241-C-112.

Tank	C-109	C-112	C-109	C-112
Analyte	%∂²(A)	%∂²(A)	%∂²(S)	%∂²(S)
ICP.acid.Al ICP.acid.Ca ICP.acid.Fe ICP.acid.Na ICP.acid.Ni ICP.acid.Pb ICP.acid.U	6% 19% 37% 100% 33% 6% 20%	8% 5% 100% 8% 6% 1% 7%	94% 81% 63% 0% 67% 94% 80%	92% 95% 0% 92% 94% 99% 93% 92%
ICP.acid.P ICP.fus.Al ICP.fus.Ca ICP.fus.Fe ICP.fus.Na ICP.fus.Pb ICP.fus.U ICP.fus.P	100% 1% 3% 6% 30% 0% 9% 65%	0% 0% 37% 1% 4% 1% 0%	99% 97% 94% 70% 100% 91% 35%	100% 100% 63% 99% 96% 99% 100%
ICP.water.Al ICP.water.Ca ICP.water.Fe ICP.water.Na ICP.water.Ni ICP.water.U ICP.water.P	NA 4% 3% 15% 7% NA 82%	30% 100% 1% 51% 1% 36% 24%	NA 96% 97% 85% 93% NA 18%	70% 0% 99% 49% 99% 64% 76%
Chloride Nitrite Nitrate Phosphate Sulphate Total cyanide	0% 41% 14% 74% 8% 0%	32% 56% 52% 27% 41% NA	100% 59% 86% 26% 92% 100%	68% 44% 48% 73% 59% NA
Uranium (µg/g) Tot.Alpha.Pu Sr-90 Pu-238 Pu-239/240 Cs-137.water Cs-137.fusion	4% 2% 5% NA 2% 22% 7%	2% NA 0% 0% 1% NA 9%	96% 98% 95% NA 98% 78% 93%	98% NA 100% 100% 99% NA 91%

Table 7-17. Relative Standard Deviations from Tanks C-112 and C-109.

	1170		-112 and			
Tank	C-109	C-112	C-109	C-112	C-109	C-112
Analyte	RSD(亨)	RSD(克)	RSD(A)	RSD(A)	RSD(S)	RSD(S)
ICP.acid.Al ICP.acid.Ca ICP.acid.Fe ICP.acid.Na ICP.acid.Ni ICP.acid.Pb ICP.acid.U	44% 17% 34% 2% 8% 78% 24%	46% 17% 10% 11% 18% 34% 40%	18% 13% 39% 13% 9% 33% 19%	23% 7% 24% 5% 8% 6% 18% 9%	76% 28% 51% 0% 12% 133% 39%	78% 30% 0% 18% 30% 59% 68% 29%
ICP.fus.A1 ICP.fus.Ca ICP.fus.Fe ICP.fus.Na ICP.fus.Pb ICP.fus.U ICP.fus.P	45% 15% 24% 8% 80% 42% 10%	46% 18% 10% 12% 39% 30% 18%	7% 4% 10% 8% 2% 21% 17%	3% 2% 11% 2% 13% 5%	78% 26% 41% 12% 113% 67% 12%	82% 32% 15% 22% 68% 52% 32%
ICP.water.Al ICP.water.Ca ICP.water.Fe ICP.water.Na ICP.water.Ni ICP.water.U ICP.water.P	NA 36% 8% 10% 41% NA 20%	33% 11% 23% 18% 24% 65% 36%	NA 12% 3% 7% 19% NA 41%	34% 58% 5% 26% 5% 74% 33%	NA 62% 14% 16% 69% NA 19%	51% 0% 41% 25% 43% 98% 59%
Chloride Nitrite Nitrate Phosphate Sulphate Total cyanide	5% 5% 10% 20% 10% 34%	15% 16% 18% 35% 17% NA	0% 6% 6% 38% 5% 2%	16% 24% 25% 34% 21% NA	8% 8% 16% 22% 18% 59%	23% 22% 24% 56% 25% NA
U (μg/g) Tot.Alpha.Pu Sr-90 Pu-238 Pu-239/240 Cs-137.water Cs-137.fusion	39% 78% 39% NA 78% 17%	52% NA 44% 110% 86% NA 5%	13% 18% 15% NA 18% 15%	11% NA 2% 6% 9% NA 2%	66% 135% 66% NA 135% 27%	78% NA 79% 137% 107% NA 7%

RSD = Relative standard deviation.

There is no significant difference between the composite sample means for 12 of 31 (39 percent) analytes and they were significantly different in 19 out of 31 (61 percent) of the analytes. The comparisons between the subsegments indicates layering within the waste for some analytes and that the waste is very heterogeneous for other analytes. These types of heterogeneity influence the magnitude of the spatial variability.

Because of the large spatial and analytical measurement variances and small df, the mean concentrations cannot be distinguished from each other. For most of the analytes, the spatial variances cannot be distinguished from each other. Although the analytical results came from the same laboratory, the analytical measurement variances were significantly different for 13 out of 28 (46 percent) analytes.

This page intentionally left blank.

8.0 CONCLUSIONS AND RECOMMENDATIONS

8.1 CONCLUSIONS FROM ANALYTICAL RESULTS

Analyses of the waste show a very small number of analytes comprising a large portion of the waste. Water is the single largest analyte, making up 38.6 percent of the total mass. There is relatively little supernatant in the tank (less than 6 percent of the total mass). Seven elements (aluminum, calcium, iron, sodium, nickel, phosphorus, and uranium) constitute approximately 25 percent of the solids mass. They also represent over 95 percent of the total cations. Two anions (NO_2 and NO_3) constitute approximately 8 percent of the solids mass. The fraction of the total anions that nitrate and nitrite represent cannot be adequately determined because the analytical method measured only soluble anions. The total cyanide content was measured and found to be less than 1.5 percent (dry basis) in each core and for the tank as a whole.

The only significant gamma emitter found in the waste was ¹³⁷Cs. Although ⁶⁰Co was also precipitated during the scavenging process, it has decayed below any level of concern and does not contribute to the heat load of the tank. No meaningful regional concentrations (hot spots) of radioisotopes or fuel were detected along the vertical axis in either core. The 137Cs concentrations were comparable between Core 47 and Core 49 and the regions with high nickel concentrations correlated with the ¹³⁷Cs activity. These observations are consistent with the historical information regarding the ferrocyanide-scavenging process and the ICP element distribution through the subsegments. The other major source of radiological activity was 90Sr, which decreased significantly as a function of depth in both Core 47 and Core 49, as expected from the fill history. Heat-load calculations are further evidence supporting the contention that the relatively high 90Sr concentrations are believed to be a phenomenon particular to the upper several inches of the waste. The bulk waste temperature in the tank, obtained from two thermocouple trees, ranges between 23° to 26 °C (74° to 78 °F). Comparisons of heat-load calculations, using the temperature profiles from the thermocouple trees and heat loads based on isotope concentrations, agree within 27 percent and are considered reasonable (Cash et al. 1993; Appendix C). The radiological activity of tank 241-C-109 waste material was relatively low (ranging from 1 to 2.5 R/hr, measured through the drill string). No significant radiological activity was found in the drainable liquid in the tank or in the water or acid digestion of the samples. This shows that 90Sr and 137Cs are quite insoluble.

Cores 47 and 49 appear to have concentrated decladding waste and hot semiworks effluent disposed on top of 1C, 1C EBs and CW that was scavenged, sometimes with ferrocyanide, sometimes not, overlying a heel bismuth phosphate first decontamination cycle waste heel. However, no firm evidence of BiPO $_4$ -1C waste was ever found from the assays. The phosphate and phosphorous profiles although suggestive, are not conclusive. The water content, particle size distribution, DSC/TGA behavior, and aluminum concentration in these two cores are different than the tank 241-C-112 core samples or the In Farm simulant behavior. First decontamination cycle and 1C EBs mixed with decladding waste rather than uranium recovered TBP waste was the primary waste that was scavenged. Profiles of 90 Sr (decreasing as a function of depth) and Cesium-137 (increasing as a function of depth) profiles agree with fill

histories. No discernable exothermic behavior was detected in these samples, even though there were measurable quantities of cyanide present. This lack of observed energetic behavior was attributed to the aluminum content of the waste; the primary reaction occurring between 260° and 300°C is believed to be the dehydration of aluminum hydroxide.

Indications from Core 48 data show that material has physical and chemical properties corresponding to those expected for ferrocyanide waste. Water content, particle size distribution, DSC/TGA behavior, nickel and total cyanide concentration are consistent with the 241-C-112 values. However, again in this case, DSC results from the suspected ferrocyanide waste in tank 241-C-109 indicate that the material is considerably less energetic than the corresponding waste simulant or the theoretical as-deposited values. Results of aging studies now underway on flowsheet simulants may demonstrate that radiolytic, hydrolytic, and thermal processes in the tanks over the last 35 years have combined to dissolve, dilute, and destroy the reactive ferrocyanide compounds. The data from tank 241-C-109 strongly indicate that the waste lacks the fuel concentration to sustain any propagating exothermic behavior and a heat source intense enough to trigger a reaction is absent.

8.2 CONCLUSIONS FROM ENERGETICS ANALYSIS

Ferrocyanide simulants made by the In Farm flowsheet are more reactive than ferrocyanide wastes derived from other processes. The waste in tank 241-C-109 and the other C Farm tanks, representing 20 to 25 percent of the ferrocyanide inventory added to the tanks, was made by a similar process and was a potential cause for concern. However, if the In Farm simulants contained at least 15 weight percent water, that moisture content precluded an uncontrolled, propagating reaction (Fauske 1992).

Three core samples were obtained from tank 241-C-109. While all three cores were broken down into smaller subsegments and examined for reactivity using DSC, none of the samples exhibited any propagating behavior. The samples had a moisture content ranging from 19 to 58 wt% water and a heat of reaction ranging from no discernable exotherm to -51.9 J/g dry material (0.0 to -12.4 cal/dry g). The onset temperatures for the endotherms were between 217° and 270°C. The onset temperatures for the observed exotherm was 272 °C, close to that predicted by the simulants. The simulant may have represented some of the waste as it was initially precipitated in the tank; several of the physical and chemical properties of the simulants are quite close to those of Core 48 waste. However, much of the waste that was scavenged was quite different from the material in tank 241-C-112. In addition, further chemical analysis indicates that the waste material has a total cyanide content much lower than expected from the simulant formulations and, correspondingly, tank 241-C-109 waste material is not as energetic as the analogous waste simulants. Tank 241-C-109 sample material is nearly 24 times less chemically reactive than the comparable In Farm simulant material (tank 241-C-109 waste: -0.05 kJ/g; In Farm 1 simulant: -1.20 kJ/g). The causes of this behavior are hypothesized to be long-term exposure to radiation fields and additional high pH cladding waste. Both of these conditions appear to degrade the ferrocyanide complexes.

Calculations of the bulk waste inventory and inventories for several analytes of interest to the various safety issues (ferrocyanide, NO_2 , NO_3 , 137 Cs, 90 Sr, plutonium, and water) were made. The calculated bulk inventory of ferrocyanide (6,800 g-mol) was far in excess of the 1,000 g-mol Ferrocyanide Watch List criteria, but the energetics results indicate that particular criteria do not account for the dispersion of the ferrocyanide in the waste (i.e., the concentration may be too low to support a self-sustaining reaction). None of the other calculated bulk inventory values exceeded any level of concern (refer to Table 8-1).

Analytical evidence from tank 241-C-109 suggests the risk from ferrocyanide compounds in Hanford Site high-level waste tanks is acceptable and that a propagating exothermic ferrocyanide reaction is incredible.

Table 8-1.	Comparison o	f Tank	241-C-109	Analyte	Values
· · · ·	to Safety				

Analyte	Safety issue criteria ¹	Calculated/ measured value
Na ₂ NiFe(CN) ₆	1,000 g-mol	6,800 g-mol
ΔH (dry basis)	-75 cal/g	-12 cal/g*
^{239/240} Pu	50 kg	3.8 kg
Temperature	300 °F (149 °C)	29 °C (85 °F)
Heat load	11.72 kw	2.85 kw
Organic content (TOC, Dry basis) (10% sodium acetate equivalent)	3.0 wt% TOC	0.45 wt% TOC

¹⁽Lindsey 1986; RHO 1988; Boyles 1992; Reep 1992). *Only observed exotherm in several measurements.

8.3 RECOMMENDATIONS

The following recommendations are made based on the data and analyses presented in this report and the goals of the characterization effort.

- The Watch List criteria for the Ferrocyanide waste tank USQ should be expanded so that it includes concentration or energetics based measurements.
- Tank 241-C-109 should be removed from the Ferrocyanide Watch List.
 If removal cannot be effected, the tank should be declared safe for
 interim-safe storage, so that routine tank farm operations can be
 performed.

- An analysis for the formate anion should be done on the water soluble material to aid in confirming that the ferrocyanide has degraded.
- In future sample analyses, the DSC assay should be performed under an inert atmosphere to better represent the waste conditions in the tank.

9.0 REFERENCES

- Abrams, R. B., 1956, *History: Metal Recovery Waste Scavenging Program*, HW-43066, General Electric Company, Richland, Washington.
- Anderson, J. D., 1990, A History of the 200 Area Tank Farms, WHC-MR-0132, Westinghouse Hanford Company, Richland, Washington.
- Babad, H., B. C. Simpson, R. J. Cash, M. A. Lilga, 1993a, *The Role of Aging in Resolving the Ferrocyanide Safety Issue*, WHC-EP-0599, Westinghouse Hanford Company, Richland, Washington.
- Babad, H., D. M. Camaioni, M. A. Lilga, W. D. Samuels, D. M. Strachan, 1993b, Tank Waste Chemistry - A New Understanding of Waste Aging, WHC-SA-1694-FP, Westinghouse Hanford Company, Richland, Washington.
- Bechtold, D. B., 1992, "Analysis of In-Plant Ferrocyanide Solids," Internal Memo 12110-PC92-014, Westinghouse Hanford Company, Richland, Washington.
- Bell, K. E., 1993, Tank Waste Remediation System Tank Waste Characterization Plan, WHC-SD-WM-PNL047, Rev. 1, Westinghouse Hanford Company, Richland, Washington.
- Bird, R. B, W. E. Stewart, E. N. Lightfoot, 1960, *Transport Phenomena*, John Wiley and Sons. New York, New York.
- Borsheim, G. L. and B. C. Simpson, 1991, An Assessment of the Inventories of the Ferrocyanide Watchlist Tanks, WHC-SD-WM-ER-133 Rev. 0, Westinghouse Hanford Company, Richland, Washington.
- Boyles, V. C., 1992, Operating Specifications for Single-Shell Waste Storage Tanks, OSD-T-151-00013 Rev D-1, Westinghouse Hanford Company, Richland, Washington.
- Brown, T. M., L. Jensen, 1993, Tank Characterization Report for Single-Shell Tank 241-U-110, WHC-EP-0643, Westinghouse Hanford Company, Richland, Washington.
- Burger, L. L., 1984, Complexant Stability Investigation, Task 1, Ferrocyanide Solids, PNL-5441, Pacific Northwest Laboratories, Richland, Washington.
- Burger, L. L., 1989, Complexant Stability Investigation, Task 1-Ferrocyanide solids, PNL-5441, Pacific Northwest Laboratory, Richland, Washington.
- Burger, L. L., and R. D. Scheele, 1990, *The Reactivity of Cs₂NiFe(CN)₆ Towards Nitrate and Nitrite Salts*, PNL-7550, Pacific Northwest Laboratory, Richland, Washington.
- Cady, H. H., 1992, Evaluation of Ferrocyanide/Nitrate Explosive Hazard, LA-12589-MS, Los Alamos National Laboratory, Los Alamos, New Mexico.

- Cash, R. J., 1993, Ferrocyanide Safety Program Removal of Six Tanks from Ferrocyanide Tank List, (letter 9259120 to J. H. Anttonen, January 14, 1993), Westinghouse Hanford Company, Richland, Washington.
- Cash, R. J., G. T. Dukelow, and C. J. Forbes, 1993, Quarterly Report on Defense Nuclear Facilities Safety Board Recommendation 90-7 for the Period Ending December 31, 1992, WHC-EP-0474-7, Westinghouse Hanford Company, Richland, Washington.
- Colby, S. A., and M. D. Crippen, 1991, *Graphical Presentation of Ferrocyanide Tank Compositions*, WHC-SA-1304-FP, Westinghouse Hanford Company, Richland, Washington.
- DOE, 1987, Final Environmental Impact Statement, Disposal of Hanford Defense High-Level Transuranic Wastes, Hanford Site, Richland, Washington, DOE/EIS-0113, Vol. 1 through 5, U.S. Department of Energy, Washington, D.C.
- Ecology, EPA, and DOE, 1992, Hanford Federal Facility Agreement and Consent Order, 2 vols., Washington State Department of Ecology, U.S. Environmental Protection Agency, U.S. Department of Energy, Olympia, Washington.
- Fauske, H. K., 1992, Adiabatic Calorimetry and Reaction Propagation Rate Tests with Synthetic Ferrocyanide Materials Including U Plant-1, U Plant-2, In Farm 1, In Farm 2 and Vendor-Procured Sodium Nickel Ferrocyanide, Fauske & Associates, Inc., Burr Ridge, Illinois.
- General Electric, 1958, *Record of Scavenged TBP Waste*, General Electric Company, Richland, Washington.
- Hanlon, B. M., 1992, Tank Farm Surveillance and Waste Status Summary Report for September 1992, WHC-EP-0182-39, Westinghouse Hanford Company, Richland, Washington.
- Harville, D. A., 1977, "Maximum Likelihood Approaches to Variance Component Estimation and to Related Problems," Journal of the American Statistical Association, p.p. 320-340.
- HASM, 1993, Single-Shell Tank Characterization Project and Safety Analysis Project Core 47, 48 and 49, Validation Report Tank 241-C-109, WHC-SD-WM-DP-036, Westinghouse Hanford Company, Richland, Washington.
- Hill, J. G., W. I. Winters, B. C. Simpson, J. W. Buck, P. J. Chamberlain, and V. L. Hunter 1991, Waste Characterization Plan for the Hanford Site Single-Shell Tanks-Appendix I: Test Plan for Sampling and Analysis of Ten Single-Shell Tanks, WHC-EP-0210 Rev. 3, Westinghouse Hanford Company, Richland, Washington.
- Hill, J. G., 1991, Modified Test Plan for the Ferrocyanide Single-Shell Tanks, 241-C-112, C-109 and T-107, Correspondence No. 915211, Westinghouse Hanford Company, Richland, Washington.

- Husa, I. E., R. E. Raymond, R. K. Welty, S. M. Griffith, B. M. Hanlon, R. R. Rios, and N. J. Vermeulen, 1993, Hanford Site Waste Storage Tank Information Notebook, WHC-EP-0625, Westinghouse Hanford Company, Richland, Washington.
- Jensen, L. and B. J. Whitcher, 1992, "Statistical Analysis of Tank 241-U-110 Data, IV: Concentration Estimates," Internal Memo 12100--PLT92-068, Westinghouse Hanford Company, Richland, Washington.
- Jeppson, D. W. and J. J. Wong, 1993, Ferrocyanide Waste Simulant Characterization, WHC-EP-0631, Westinghouse Hanford Company, Richland, Washington.
- Jungfleisch, F. M., 1984, TRAC: A Preliminary Estimation of the Waste Inventories in Hanford Tanks Through 1980, SD-WM-TI-057, Rockwell Hanford Operations, Richland, Washington.
- Lilga, M. A., M. R. Lumetta, W. F. Riemath, R. A. Romine, and G. F. Schiefelbein, 1992, "Ferrocyanide Safety Project, Subtask 3.4, Aging Studies FY 1992, Annual Report," PNL-8387 UC-721, Pacific Northwest Laboratory, Richland, Washington.
- Lindsey, D. W., 1986, Operating Specifications for Single-Shell Waste Storage Tanks, OSD-T-151-00013, Rockwell Hanford Operations, Richland, Washington.
- National Defense Authorization Act for Fiscal Year 1991, Public Law 101-510, Section 3137, "Safety Measures for Waste Tanks at Hanford Nuclear Reservation," November 5, 1990.
- Peach, J. D., 1990, "Consequences of Explosion of Hanford's Single-Shell Tanks Are Understated," (Letter B-241479 to C. M. Synar, Chairman of Environment, Energy and Natural Resources Subcommittee, Committee on Government Operations, House of Representatives), GAO/RCED-91-34, General Accounting Office, Washington, D.C.
- Pederson, L. R., H. Babad, S. A. Bryan, A. J. Schmidt, 1993, Autogeneous Hydrothermal Oxidation in Simulated Hanford Double-Shell Tank Wastes, PNL-SA-22181, Pacific Northwest Laboratory, Richland, Washington.
- Petersen, R. G., 1985, Design and Analysis of Experiments, First Edition, Marcel Dekker, Inc., New York, New York.
- Reep, I. E., 1992, Status Report on Resolution of Waste Tank Safety Issues at the Hanford Site, WHC-EP-0600, Westinghouse Hanford Company, Richland, Washington.
- RHO, 1988, Criticality Prevention Specifications for Waste Storage Tanks and Associated Equipment, CPS-T-149-00010, Rockwell Hanford Operations, Richland, Washington.

- Scheele, R. D., L. L. Burger, J. M. Tingey, S. A. Bryan, G. L. Borsheim, B. C. Simpson, R. J. Cash, and H. H. Cady, 1991, "Ferrocyanide-Containing Waste Tanks: Ferrocyanide Chemistry and Reactivity," in the Proceedings of Environmental Restoration 91, University of Arizona, Tucson, Arizona.
- Schmidt, W. C., and M. J. Stedwell, 1954, *Production Test 221-T-18 Scavenging of First Cycle Waste*, HW-33252, General Electric Company, Richland, Washington.
- Schneider, K. J. 1951, Flow Sheets and Flow Diagrams of Precipitation Separations Process, HW-23043, General Electric Company, Richland, Washington.
- Simiele, C. J., 1991, Single-Shell Tank Phase 1A/1B Procedure Compendium, WHC-MR-0213, Westinghouse Hanford Company, Richland, Washington.
- Sloat, R. J., 1954, TBP Plant Nickel Ferrocyanide Scavenging Flowsheet, HW-30399, General Electric Company, Richland, Washington.
- Sloat, R. J., 1955, In Farm Scavenging Operating Procedure and Control Data, HW-38955 Rev. 1, General Electric Company, Richland, Washington.
- Simpson, B. C., G. L. Borsheim, L. Jensen, 1993, Tank Characterization Data Report: Tank 241-C-112, WHC-EP-0640, Westinghouse Hanford Company, Richland, Washington.
- Snedecor, G. W., and W. G. Cochran, 1980, Statistical Methods, Seventh Edition, Iowa State University Press, Ames, Iowa.
- Wagner, 1992, Hanford Waste Vitrification Plant Feed Characterization Requirements, WHC-SD-HWV-SM-001, Rev. 4, Westinghouse Hanford Company, Richland, Washington.
- Weast, R., 1984, Handbook of Chemistry and Physics, 64th Ed., CRC Press, Boca Raton, Florida.
- Winters, W. I., L. Jensen, L. M. Sasaki, R. L. Weiss, J. F. Keller, A. J. Schmidt, and M. G. Woodruff 1990a, Waste Characterization Plan for the Hanford Site Single-Shell Tanks, WHC-EP-0210 Rev. 1, Westinghouse Hanford Company, Richland, Washington.
- Winters, W. I., L. Jensen, L. M. Sasaki, R. L. Weiss, J. F. Keller, A. J. Schmidt, and M. G. Woodruff 1990b, Waste Characterization Plan for the Hanford Site Single-Shell Tanks, WHC-EP-0210 Rev. 2, Westinghouse Hanford Company, Richland, Washington.
- Wodrich, D. D., G. S. Barney, G. L. Borsheim, D. L. Becker, W. C. Carlos, M. J. Klem, J. L. Ryan, R. E. Van der Cook, 1992, Summary of Single-Shell Waste Tank Stability, WHC-EP-0347 Supplement, Westinghouse Hanford Company, Richland, Washington.

APPENDIX A

CALCULATIONS

This page intentionally left blank.

APPENDIX A: CALCULATIONS

Theoretical Energetic Behavior of C-109 Waste:

Assume 1.0 and 1.5 vol% precipitate formation; use waste volumes generated from Borsheim-Simpson spreadsheet model.

Ferrocyanide waste volume: @ 1.0 vol% = 25,000 gallons

0.1.5 vol% = 37,500 gallons

Input mole of ferrocyanide: 47,300 g-mol Na₂NiFe(CN)₆

Calculated moles

ferrocyanide remaining: @ 1.0 and 1.5 vol% = 47,300 g-mol Na₂NiFe(CN)₆

Analytical estimate 38,400 g-mol Na₂NiFe(CN)₆ derived from Ni content (discounting Ni from ⁶⁰Co scavenging, and using the acid ICP composite values)

(14,033 μ g Ni/wet g)*(1 g/1E+06 μ g)*(g-mol Ni/58 g)*(284.6 Mg)=68,900 g-mol Ni

Ni from non-FeCN scavenging: 30,500 g-mol; thus 68,900-30,500=38,400 g-mol Ni and (38,400 g-mol Ni)*(1 g-mol Na₂NiFe(CN)₆/g-mol Ni)=38,400 g-mol Na₂NiFe(CN)₆

Bulk Mass of Ferrocyanide waste (Density = 1.20 g/mL):

(25,000 gal)*(3785 mL/gal)*(1.20 g/mL) = 1.14E+08 g waste(37,500 gal)*(3785 mL/gal)*(1.20 g/mL) = 1.70E+08 g waste

Mass of Ferrocyanide in Ferrocyanide waste:

 $(47,300 \text{ g-mol})*(316.7 \text{ g/g-mol}) = 1.50E+07 \text{ g Na}_2\text{NiFe(CN)}_6$

Weight Percent Ferrocyanide in waste (range):

@ 1.0 vol% : $1.50E+07 \text{ g Na}_2\text{NiFe}(CN)_6*(100) = 13.16 \text{ wt}$ %

1.14E+08 g waste

0.1.5 vol%: $1.50E+07 \text{ q Na}_2\text{NiFe(CN)}_6*(100) = 8.82 \text{ wt}\%$

1.70E+08 q waste

Assume 37% average water content, 63% solids (Average of Core 47 and Core 49 TGA measurements)

Weight Percent Ferrocyanide (Dry basis)

@ 1.0 vol% : 1.50E+07 g $Na_2NiFe(CN)_6*(100) = 20.88$ wt%

1.14E+08 g waste (0.63)

@ 1.5 vol% : $1.50E+07 \text{ q Na}_2\text{NiFe}(CN)_6*(100) = 14.00 \text{ wt}$ %

1.70E+08 g waste (0.63)

Simulant	Ferrocyanide Content (Dry basis)	ΔΗ _{simulant} (kJ/g dry simulant)	ΔΗ _{ferrocyanide} (kJ/g Na ₂ Nife(CN) ₆)
U Plant-l	4.3 wt%	-0.17	-3.95
U Plant-2	8.6 wt%	-0.34	-3.95
In Farm-1	25.5 wt%	-1.20	-4.71

Table A-1. Energetic Properties of Waste Simulants (Fauske 1992).

A direct, linear relationship can be assumed between the ferrocyanide (or cyanide) content of the waste and the energy content of the material.

 $\Delta H_{sample} = (Wt\% Na_2NiFe(CN)_6)*(-3.95 KJ/g Na_2NiFe(CN)_6)$

Therefore, the theoretical energetic behavior of the waste as it was initially stored in tank 241-C-109 is between:

(14.00%)*(-3.95) = -0.55 KJ/g waste; or 132 cal/g dry waste

(20.88%)*(-3.95) = -0.82 KJ/g waste; or 197 cal/g dry waste

The basis for the exothermic potential of the reaction is $-3.95 \ \text{KJ/g}$ $\text{Na}_2\text{NiFe}(\text{CN})_6$. This value is used for several reasons. The measurement of the two U Plant simulants was performed on much larger samples, thus is not as sensitive to rounding and experimental error. In addition, the measured gaseous products from the U Plant reaction were 0.075 moles produced out of a theoretical 0.11 moles; this represents a release fraction of 68.2%, suggesting incomplete or less efficient combustion. There are several alternative reaction paths, however, the most reactive thermodynamic pathway has a ΔH of $-9.6 \ \text{KJ/g} \ \text{Na}_2\text{NiFe}(\text{CN})_6$. Therefore a reaction efficiency for this waste matrix is (-3.95/-9.6)*100 = 41.1%. It can be assumed that the release fraction and reaction efficiency in the waste matrix would not be any greater than that exhibited by the simulants. However, greater heats of reaction can be expected from mixtures with higher fuel contents, as long as there is sufficient oxidizer. This behavior is expected because factors contributing to combustion efficiency are more favorable: (1) a higher fuel/oxidant interface and (2) fewer solid diluents. This may be why the In Farm-1 simulant exhibits a higher heat of reaction than the U Plant simulants.

Determination of 137Cs values from Revised Borsheim/Simpson Model:

The approximate amount of 137 Cs processed through 241-C-109 during the scavenging campaign was 319,000 Ci. The half-life of 137 Cs is 30.17 years. The decay period for the waste is assumed to be 35 years (1958-1993). The decay factor for this timespan is thus:

 $e^{-(\ln 2/30.17)*35} = 0.4475$

Therefore, the 137 Cs remaining from the scavenging campaign is (319,000 Ci)*(0.4475) = 142,800 Ci

The amount of ¹³⁷Cs captured in the tank is assumed to be directly proportional to the amount of ferrocyanide waste solids retained, however in each case, there were no transfers of solids out of the tank. Therefore, the inventory of ¹³⁷Cs remaining from the scavenging campaign does not change.

Comparison of Actual Waste Values with Theoretical Estimates

Total cyanide values can provide estimates of ferrocyanide content; and this derived ferrocyanide content can be used to determine the energy content of the sample (assuming all cyanide detected is present as ferrocyanide).

```
\frac{(316.7 \text{ g/g-mol Na_NiFe(CN)}_6)}{(156 \text{ g CN/g-mol Na_NiFe(CN)}_6)}*(Wt\% \text{ cyanide}) = Wt\% \text{ Na_NiFe(CN)}_6
```

and using the derived ferrocyanide value in the previously described energetic relationship:

$$\Delta H_{sample} = (Wt\% Na_2NiFe(CN)_6)*(-3.95 KJ/g Na_2NiFe(CN)_6)$$
 (1)

Conversely, the energy content of the waste is a reasonable indicator of the ferrocyanide content in the waste. By rearranging Equation (1)

$$\frac{\Delta H_{\text{paralle}}}{-3.95 \text{ KJ/g}} \frac{\Delta H_{\text{paralle}}}{\text{Na}_{2}\text{NiFe(CN)}_{6}} = \text{Wt% Na}_{2}\text{NiFe(CN)}_{6}$$

However, because of the semi-quantitative nature of the DSC assay, values obtained in this manner are likely no better than qualitative results (±100%) for low fuel concentrations/small exotherms. For samples with high fuel contents (and consequently, exhibiting strong exotherms), the values are considered much more accurate. In this case, the samples are exhibiting strong endothermic behavior making a comparison even more difficult. However, even with the endotherms observed, if the fuel concentration was present at its theoretical level, exotherms would be observed.

Calculation of Bulk Inventories for Analytes of Importance

From the distribution of the solids and liquids in the samples, the measurements from tank farm surveillance, and the analyte profiles from the quarter-segments, an overall tank profile can be visualized (Figure A-1). These observations from the tank suggest that there are distinct regions in the tank, each possessing a specific volume and characteristic compositions. For the first interpretation, these regions are:

- Dished Bottom: First decontamination cycle BiPO₄ waste or unscavenged Uranium Recovery (UR) waste.
- Tank Layer 1: Unscavenged UR waste and Ferrocyanide-scavenged Uranium Recovery (FeCN) waste.
- Tank Layer 2: Ferrocyanide-scavenged supernates from BY Farm scavenged for 90Sr and 60Co without ferrocyanide.

- Tank Layer 3: Ferrocyanide-scavenged waste, plus Coating Waste, Hot Semiworks, and 1C Evaporator Bottoms.
- Supernatant.

The volumes for each region were derived as follows:

• Dished Bottom: 10,000 gallons (39,000 L)

This is the volume of waste recorded in the dish at the time prior to scavenging.

• Tank Layer 1: 19,800 gallons (75,000 L)

Calculate a volume using a slab 1.5 subsegments high. (2,750 gallons/inch)*(4.75 in./subsegment)*[0.5*(1.5+0 subsegments)] = 19,600 gallons. Compare with model estimate at 1.5 vol% = 19,840 gallons; use higher value (3 sig. figures).

• Tank Layer 2: 10,300 gallons (39,000 L)

Estimate derived from two batches of nonferrocyanide scavenged solids using a model value of 1.5 vol% solid formation.

• Tank Layer 3: 21,900 gallons (83,000 L)

Three batches of ferrocyanide scavenged solids using model estimates of 1.5 vol% solid formation (17,600 gallons) plus residual solids from additional waste transfers. Extreme ⁹⁰Sr values are only found in upper subsegment (HS discharge). In addition, large amounts of transferred waste that had solids settled out prior to their introduction were also transferred to the tank. The ⁹⁰Sr-containing volume was calculated using an estimated 2.5 vol% solids (3,300 gal) and the supernatant as having an estimated 0.125 vol% solids (1,000 gal).

Total Solids Volume = 62,000 gallons (235,000 L)

• Supernatant: 4,500 gallons (17,000 L)

Calculate a volume using a slab 1.1 inches high on the Core 47 side and 2.2 inches high on the Core 49 side. (2,750 gallons/inch)*(1.1 in.)+(0.5)*(2,750 gallons)*(1.1 in.) = 4,500 gallons.

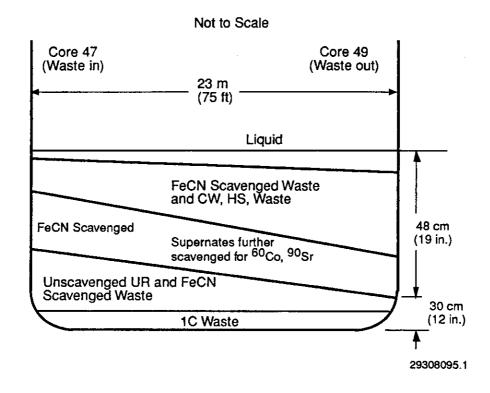
Total Waste Volume: 66,500 gallons

Check calculated volume with tank surveillance records: 66,000 gallons

Percent Difference = $\frac{66.500 - 66.000*100 = +0.8\%}{66.000}$

Using these volumes and a bulk density of 1.20 g/mL for the solids and 1.10 g/mL for the supernatant, masses for the various regions can be calculated. Also, representative concentrations for the various analytes from each of the regions can be developed.

Figure A-1. Waste Profile of Tank 241-C-109.



- Dished Bottom: First decontamination cycle BiPO₄ waste or unscavenged Uranium Recovery (UR) waste.
- Tank Layer 1: Unscavenged and Ferrocyanide-scavenged Uranium Recovery (FeCN) waste.
- Tank Layer 2: Scavenged supernates from BY Farm scavenged for ⁹⁰Sr and ⁶⁰Co without ferrocyanide.
- Tank Layer 3: Ferrocyanide scavenging waste, plus Coating Waste, Hot Semiworks, and 1C Evaporator Bottoms.
- Supernatant.

Tank Region	Volume (gallons)	Mass (Mg)	Concentration Values Used for Inventory Estimate
Dished Bottom	10,000	46.8	Core 47-1D
Tank Layer 1	19,800	90.0	Avg. Core 48-1C and 48-1D
Tank Layer 2	10,300	44.9	Avg. Core 47-1C and 49-1C and 49-1D
Tank Layer 3	21,900	95.5	Avg. Core 47-1B and Core 49-1B
Supernatant	4,500	18.7	Liquid Composite
Total	66,500	303.3	

Table A-2. Basis for Bulk Inventory Calculations.

Tables A-3 through A-7 show the concentration values used to calculate the inventory estimates and the calculated bulk inventories for selected analytes.

Compare ICP water leach phosphorous value with IC phosphate value.

Formula weights:

P: 31; PO₄³⁻: 95

Multiplier to

convert P to PO₄:

 $\frac{95 \text{ q PO}/3^{-}/\text{q-mol}}{31 \text{ g P/g-mol}} = 3.1$

Since phosphates were used extensively in waste processing, assume all insoluble P is present as $P0_4^{3-}$; subtract fusion ICP P value and add corrected amount to $P0_4^{3-}$.

Phosphorous Percent Soluble:

Core
$$47--\underline{6,990*100}_{19,900} = 35.1\%$$

Core
$$48 - 8.690 \times 100 = 43.0\%$$

20,200

Core
$$49 - \frac{4,160*100}{14,600} = 28.5\%$$

Table A-3. Bulk Inventory Basis for Energetics Analytes.

Region	Volume	Mass	TOC	Total CN	NO2	NO3	H2O
	(L)	(g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)
Dish	39000	46800000	2200	5800	40000	38500	394000
Layer 1	75000	97500000	3600	10000	51000	54800	522000
Layer 2	39000	44850000	2300	6000	42000	40300	354000
Layer 3	83000	95450000	2000	3300	27000	26700	195000
Supernatant	17000	18700000	2600	5400	71000	72000	707000
Totals	253000	303300000					

Table A-4. Calculated Inventories for Energetic Analytes.

Region	Volume	Mass	TOC	Total CN	NO2	NO3	H2O
	(L)	(g)	(g)	(g)	(g)	(g)	(g)
Dish	39000	46800000	102960	164493	1872000	1801800	18439200
Layer 1	75000	97500000	351000	466050	4972500	5343000	50895000
Layer 2	39000	44850000	103155	173839	1883700	1807455	15876900
Layer 3	83000	95450000	190900	253563	2577150	2548515	18612750
Supernatant	17000	18700000	48620	29587	1327700	1346400	13220900
Mass Wet Solids	236000	2.85E+08	7.48E+05	1.06E+06	1.13E+07	1.15E+07	1.04E+08
Wt%			0.26	0.37	3.97	4.04	36.48
Total Content	253000	303300000	7.97E+05	1.09E+06	1.26E+07	1.28E+07	1.17E+08
Wt% (wet)			0.26	0.36	4.17	4.24	38.59

Table A-5. Bulk Inventory Basis for Cesium-137 and Strontium-90.

Region	Volume (L)	Mass (g)	Cs-137 (uCi/g)	Sr-90 (uCi/g)	Cs-137 (Ci)	Sr-90 (Ci)	
Dish	39000	46800000	950	200	44460	9360	
Layer 1	75000	97500000	1190	135	116025	13163	
Layer 2	39000	44850000	610	290	27359	13007	
Layer 3	83000	95450000	230	2400	21954	229080	
Supernatant	17000	18700000	5.6	0.1	105	2	
Totals	253000	303300000			209901.7	264610.8	
Heat Load (w)					990.7361	1772.892	2763.62894
Heat Load (BTU/hr))				3379.417	6059.588	9439.00661

(In Layer 3, only the value from Core 49 was used—the partition of the core into subsegments grossly distorted the concentration after homogenization)

Compare values with method using an average of the core composite values

Cs-137 (0.082 Ci/g)*(285,000,000 g) = 233,700 Ci Sr-90

 $(0.077 \text{ Ci/g})^*(285,000,000 \text{ g}) = 218,500 \text{ Ci}$

WHC-EP-0668

Region	Volume	Mass	Al	Ca	Fe	Na	Ni	P	Pb	Si	U
	(L)	(g)	(ug/g)								
Dish	39000	46800000	32000	28000	15300	102600	25600	30100	14300	22200	5800
Layer 1	75000	97500000	8600	23000	21000	108900	34000	22100	600	2600	15600
Layer 2	39000	44850000	95700	19700	13600	72500	28400	14800	1300	2900	6600
Layer 3	83000	95450000	158500	7400	39500	47000	15100	5900	3500	10800	9900
Supernatant	17000	18700000	160	210	1680	96900	340	4200	0	70	0
Totals	253000	303300000									

Table A-7.

Calculated Inventories for ICP Analyses.

Region	Volume	Mass	Al	Ca	Fe	Na	Ni	P	Pb	Si	U
	(L)	(g)	(g)	(g)	(g)	(g)	(g)				
Dish	39000	46800000	1497600	1310400	716040	4801680	1198080	1408680	669240	1038960	271440
Layer 1	75000	97500000	838500	2242500	2047500	10617750	3315000	2154750	58500	253500	1521000
Layer 2	39000	44850000	4292145	883545	609960	3251625	1273740	663780	58305	130065	296010
Layer 3	83000	95450000	15128825	706330	3770275	4486150	1441295	563155	334075	1030860	944955
Supernatant	17000	18700000	2992	3927	31416	1812030	6358	78540	0	1309	0
Mass Wet Solids		2.85E+08	2.18E+07	5.14E+06	7.14E+06	2.32E+07	7.23E+06	4.79E+06	1.12E+06	2.45E+06	3.03E+06
Wt%			7.64	1.81	2.51	8.14	2.54	1.68	0.39	0.86	1.07

APP A-11

Table A-8. Comparison of Water Leach ICP Phosphorous Value with IC Phosphate Value.

Sample	Water Leach P ICP value (#g/g)	Converted PO ₄ value derived from ICP (µg/g)	IC PO ₄ 3- Value (µg/g)	Percent Difference
Core 47	6,990	21,420	22,100	3.2
Core 48	8,690	26,630	26,700	0.30
Core 49	4,160	12,750	12,800	0.40

To correct mass balance for unanalyzed hydroxide content, multiply analyte concentration by multiplier.

Other multipliers:

Aluminum; assume aluminum is present predominantly as Al(OH)3.

Formula weights:

A1: 27; A1(OH)₃: 78

Multiplier to

convert Al to Al(OH)₃: $\frac{78 \text{ q Al}(OH)_3/q-\text{mol}}{27 \text{ g Al}/q-\text{mol}} = 2.9$

Iron; assume iron is present predominantly as Fe(OH)₂.

Formula weights:

Fe: 56; Fe(OH)₂: 90

Multiplier to

convert Fe to Fe(OH)₂: $\frac{90 \text{ q Fe(OH)}_2/\text{q-mol}}{56 \text{ q Fe/q-mol}} = 1.6$

Nickel; assume nickel is present predominantly as Ni(OH)2.

Formula weights:

Ni: 58; Ni(OH)₂: 92

Multiplier to

convert Ni to Ni(OH)₂: $\frac{92 \text{ q Ni(OH)}_2/\text{q-mol}}{58 \text{ g Ni/g-mol}} = 1.6$

Uranium; assume uranium is present predominantly as $UO_2(OH)_2$.

Formula weights:

U: 238; $UO_2(OH)_2$: 304

Multiplier to

convert U to $UO_2(OH)_2$: $\frac{304 \text{ g } \text{UO}_2(\text{OH})_2/\text{q-mo}}{238 \text{ g } \text{U/g-mo}} = 1.3$

Total Carbon; assume carbon is present in three forms, and that the TOC and TIC assays did not consume or measure any cyanide:

Organic carbon (as acetate): $C_2H_3O_2$ Inorganic carbon (as carbonate): CO_3^{2-2}

Formula weights: C: 12; $C_2H_3O_2^-$: 59; CO_3^{2-} : 60

Multiplier to

convert TOC to acetate: $\frac{59 \text{ q } C_2H_3O_2^{-}/\text{q-mol}}{24 \text{ g } C/\text{g-mol}} = 2.45$

Multiplier to convert

TIC to carbonate: $\frac{60 \text{ g CO}_{32}/\text{q-mol}}{12 \text{ g C/g-mol}} = 5$

Potential endotherm in 241-C-109:

Consider the reaction $2A1(OH)_3 ----> A1_2O_3 + 3H_2O_3$

 ΔH_{\star} (kcal/mol) 2(-304.2) -407.95 3(-32.57)

thus, the heat of reaction, $\Delta H_{rxn} = \Delta H_{f products} - \Delta H_{f reactants}$

= (-407.95) + (-97.71) - (-608.4)= +102.74 kcal

Therefore, ΔH for each g-mol Al(OH)₃ is 102.74/2 or 51.37 kcal/g-mol Al(OH)₃.

Now, at 100,000 μ g Al/g wet sample = 10 wt% Al

(0.1 g Al/g wet sample)*(g-mol Al(OH) $_3$ /27 g Al)*(51.37 kcal/g-mol Al(OH) $_3$)* (4181 J/kcal) = 795 J/g wet sample @ 10 wt% Al

The degree of agreement between the calculated and average observed endotherms indicates that they are not solely attributable to the decomposition of aluminum hydroxide. However, it appears that this reaction may contribute significantly to the observed endothermic behavior.

Calculated A1(OH)3 Avg. Measured Theoretical Sample Wt% Al Endotherm Heat of Reaction % Total (J/g)Endotherm (J/g)47-1B 13.2 1049 1555 67.5 47-1C 12.0 954 610 156.4 47-1D 3.2 254 508 50.0 47-Comp. 11.7 930 1084 85.8 49-1B 18.5 1471 2188 67.2 49-1C 9.6 763 565 135.0 49-1D 7.1 564 305 184.9 12.7 49-Comp. 1010 922 109.5

Table A-9. Energetic Calculations.

Typical process stream calculations:

Coating Waste: Calculate wt% (μ g/g) composition of Al, Si, and U. composition given on pg. 8 of HW-23043 and reduce water content.

7,900 lbs total 1,746 lbs solute 6,154 lbs water

638 lbs NaAlO₂ (in solute)

1 lb uranium

Assume $NaA10_2$ ---> $A1(OH)_3$ (s); $NaSiO_3$ precipitates as well, other compounds remain in solution.

Dry solids mass = 638 lbs $NaAlO_2*78$ lb $Al(OH)_3/mol$ + 27 lb $NaSiO_3$ = 633.9 lb 82 lb $NaAlO_2/mol$

Analyte mass: 27 lb A1/78 lb A1(OH)₃*606.9 lbs A1(OH)₃ = 210 lbs A1 28 lb Si/99 lb NaSiO₃*27 lb NaSiO₃ = 7.7 lb Si

Borsheim-Simpson Model Run at 1.0 vol%.

TANK-C109 ASSUME 1% SLUDGE FORMATION

DATE	TANK CHG.	SUPERNATE (GAL)	SLUDGE (GAL)	TRANSFER AMOUNT	SUPERNAT AMT, IN TANK	SUPERNAT	GAL. SLUDGE TRANS.	GAL. SUPER. REMAIN.	GAL. SLUDGE REMAIN.	TOTAL WASTE REMAIN.	MOLES Fe(CN)6 REMAIN.	MOLES Fe(CN)6 PRECIP'D	MOLES Fe(CN)6 TRANS.	DISPOSAL LOCATION
INIT.	148831	138831	10000	0	138831	0	0	138831	10000	148831	0	0	0	C109
2-1956-1	384206	380364	3842	437913	519195	437913	0	81282	13842	95124	7271	7271	0	BC-4
4-1956-1	449618	445122	4496	488176	526404	488176	0	38228	18338	56566	11526	4255	0	BC-9
8-1956-3	319483	316288	3195	0	354516	0	0	354516	21533	376049	17572	6046	0	
10-1957-2	169381	167687	1694	455814	522203	455814	0	66389	23227	89616	20777	3206	0	BC-15
13-1957-2	451683	4471 6 6	4517	462700	513555	462700	0	50855	27744	78599	20777	0	C	BC-17
16-1957-3	234104	231763	2341	245121	282618	245121	0	37497	30085	67582	20777	0	0	BC-6
19-1957-3	473717	468980	4737	446175	506477	446175	0	60302	34822	95124	29742	8965	0	BC-20
23-1 95 7-3	253383	250849	2534	256138	311151	256138	0	55013	37356	92369	34538	4795	0	BC-21
27-1957-4	448929	444440	4489	432404	499453	432404	0	67049	41845	108894	47282	12744	0	BC-22
TOTALS	3333335	3291490	41845	3224441	4174404	3224441	0	67049	41845	108894	47282	47282	0	
		TOTAL	LIQUID	SOLID										
TANK VOL	(GAL)⊭	108894	67049	41845	•									
Tank Ht.(FT) =	3.9	2.0	1.9										

TANK-C109 POST-1957 TRANSFERS (1% BASIS)

			,, ,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,									
DATE	TANK CHG. (GAL)	TRANSFER FROM	SUPERNAT (GAL)	SLUDGE (GAL)	TRANSFER AMOUNT	SUPERNAT AMT. IN TANK	GALLONS SUPER. TRANS.	GAL. SLUDGE TRANS.	GAL. SUPER. REMAIN.	GAL. SLUDGE REMAIN.		1
 1957-4	108894		67049	41845		67049	\$	0	67049	41845	108894.0	
1959-2	261000	105C	260674	326		327723		0	327723	42171	369894.0	
1959-3	154000	105C	153808	193		481530		0	481530	42364	523894.0	+
1960-4	6000	LINE	6000			487530		0	487530	42364	529894.0	
1962-2	21000	HS	20475	525	137000	508005	137000	0	371005	42889	413894.0	
1962-4	58000	HS	56550	1450		427555		0	427555	44339	471894.0	
1964-2	35000	HS	34125	875		461680		0	461680	45214	506894.0	
1965-2	19000	HS	18525	475		480205		0	480205	45689	525894.0	
1966-2	13000	108C	12984	16		493189		0	493189	45705	538894.0	
1970-1	19000	203C	18976	24	397000	512165	397000	0	115165	45729	160894.0	
1970–2	375000	110C	374531	469		489697		0	489697	46198	535894.0	
1975–3	0				364000	489697	364000	0	125697	46198	171894.0	
1976-1	0	LEVEL ADJ.	•		121000	125697	121000	0	4697	46198	50894.0	
1976-1	END OF MOD	DELINGTANK	REMOVED FR	OM SERVIC	Έ		•					

	TOTAL	LIQUIDS	SOLIDS
TANK VOL (GAL)=	50894.0	4696.5	46197.5
Tank Ht.(FT)=	2.2	0.1	2.0

TANK-C109

TANK VOL (GAL)≖ Tank Ht.(FT)≔

108894 3.9

51126 1.5

57768 2.4

	ASSUME 1.5	5% SLUDGE FO	RMATION											
DATE	TANK CHG. (GAL)	SUPERNATE (GAL)	SLUDGE (GAL)	TRANSFER AMOUNT	SUPERNAT AMT. IN TANK	GALLONS SUPERNAT TRANS.	GAL. SLUDGE TRANS.	GAL. SUPER. REMAIN.	GAL. SLUDGE REMAIN.	TOTAL WASTE REMAIN.	MOLES Fe(CN)6 REMAIN.	MOLES Fe(CN)6 PRECIP'D	MOLES Fe(CN)6 TRANS.	DISPOSAL LOCATION
INIT.	148831	138831	10000	0	138831	0	0	138831	10000	148831	0	0	0	C109
2-1956-1	384206	378443	5763	437913	517274	437913 -	0	79361	15763	95124	7271	7271	0	BC-4
4-1956-1	449618	442874	6744	488176	522235	488176	0	34059	22507	56566	11526	4255	0	BC-9
8-1956-3	319483	314691	4792	0	348749	0	0	348749	27300	376049	17572	6046	0	
10-1957-2	169381	166840	2541	455814	515590	455814	0	59776	29840	89616	20777	3206	0	BC-15
13-1957-2	451683	444908	6775	462700	504683	462700	0	41963	36616	78599	20777	0	0	BC-17
16-1957-3	234104	230592	3512	245121	272576	245121	0	27455	40127	67582	20777	0	0	BC-6
19-1957-3	473717	466611	7106	448175	494066	446175	0	47891	47233	95124	29742	8965	0	BC-20
23-1957-3	253383	249582	3801	256138	297473	256138	0	41335	51034	92369	34538	4795	0	BC-21
27-1957-4	448929	442195	6734	432404	483530	432404	0	51126	57768	108894	47282	12744	0	BC-22
TOTALS	3333335	3275567	57768	3224441	4095008	3224441	0	51126	57768	108894	47282	47282	0	
		TOTAL	LIQUID	SOLID										
TANK VOL ((GAL)≖	108894	51126	57768										
Tank Ht./FT)==	3.9	1.5	2.4										

TANK-C109 POST-1957 TRANSFERS (1.5% BASIS)

DATE	TANK CHG. (GAL)	TRANSFER FROM	SUPERNAT (GAL)	SLUDGE (GAL)	TRANSFER AMOUNT	SUPERNAT AMT. IN TANK	GALLONS SUPER. TRANS.	GAL. SLUDGE TRANS.	GAL. SUPER. REMAIN.	GAL. SLUDGE REMAIN.		Borshei
1957-4	108894		51126	57768		51126		0	51126	57768	108894.0	
1959–2	261000		260674	326		311800		0	311800	58094	369894.0	Si
1959–3	154000		153808	193		465607		0	465607	58287	523894.0	哥
1960-4	6000		6000			471607		0	471607	58287	529894.0	
1962-2	21000		20475	525	137000	492082	137000	0	355082	58812	413894.0	
1962-4	58000	HS	56550	1450		411632		0	411632	60262	471894.0	, Ş
1964-2	35000		34125	875		445757		0	445757	61137	506894.0	
1965-2	19000		18525	475		464282		0	464282	61612	525894.0	, –
1966-2	1,3000		12984	16		477266		0	477266	61628	538894.0	Run
1970-1	19000		18976	- 24	397000	496242	397000	0	99242	61652	160894.0	, 3
1970-2	375000	110C	374531	469		473774		0	473774	62121	535894.0) A
1975–3	0				364000	473774	364000	0	109774	62121	171894.0	-
1976-1		LEVEL ADJ.			105000	109774	105000	0	4774	62121	66894.0	்
1976–1	END OF MOD	ELINGTANK		IOM SERVIO	SOLIDS							۷01% ,
TANK VO	• •		66894.0	4773.5	62120.5							SO
Tank Ht.(I	FT)=		2.6	0.1	2.5							Post-Scavenging

	TOTAL	LIQUIDS	SOLIDS
TANK VOL (GAL)=	66894.0	4773.5	62120.5
Tank Ht.(FT)=	2.6	0.1	2.5

Table A-14. Typical Analyte Concentrations in Precipitated Coating
Waste

Analyte	Mass (1bs)	Dry Wt%	Concentration (µg/dry g)	Concentration 0 40% water (µg/g)	Concentration @ 50% water (µg/g)
ΑΊ	210	33.1	331,000	198,600	165,500
Si	7.66	1.21	12,100	7,260	6,050
U	1	0.16	1,600	960	800

Neutralized 1C Waste: Calculate wt% ($\mu g/g$) composition of Bi, Si, F, and U; use composition given on pg. 32 of HW-23043.

18,500 lbs total 3,087 lbs solute

15,413 lbs water 3.8 lbs U

44 lbs Bi 73.8 lbs SiF₆²⁻

802.5 lbs Na 1,579 lbs NO

Assume NO_3 is present as $NaNO_3$, thus $\frac{1.579.5 \text{ lbs} \pm 453.6 \text{ g/lb}}{2,040 \text{ gal} \pm 3.785 \text{ L/gal} \pm 62 \text{ g/mol}} = 1.5 \text{ M} \text{ NO}_3$

1.5 M is not near saturation; water and nitrate do not precipitate.

The amount of sodium that precipitates is that not associated with the nitrate: Total moles of sodium = 802.5 lbs/23 lb/mol or 34.9 lb-mol

 $\frac{1.579.5 \text{ lbs NO}_3}{62 \text{ lbs/mol}} = 25.5 \text{ lb-mol nitrate (and associated sodium)}$

Mass of sodium nitrate in solution: 25.5 lb-mol*85 lb/lb-mol = 2,165 lbs.

Therefore, $(34.9-25.5 \text{ lb-mol})*23 \text{ lb Na/lb-mol} = 216.7 \text{ lbs Na precipitate and 585.8 lb Na remain in solution. If everything but the NaNO₃ and water precipitate, then the mass of dry solids = 18,500 lbs - <math>(15,413+2,165)$ = 922 lbs.

Table A-15. Typical Analyte Concentrations in Precipitated 1C Waste.

Analyte	Mass	Dry Wt%	Dry Concentration (#g/g)	Concentration @ 50 water (µg/wet g)	Concentration @ 40% water (µg/wet g)
Bi	44	4.77	47,700	23,850	28,620
Si	14.6	1.58	15,800	7,900	9,480
F	59.2	6.42	64,200	32,100	38,520
U	3.8	0.41	4,100	2,050	2,460

UR waste: Calculate wt% ($\mu g/g$) composition of U at a density of 1.3. Use flowsheet composition given on pg. 5 of HW-30399. Uranium concentrations will range from 0.007 to 0.14 M (flowsheet concentrations to 20x flowsheet) or approximately 1.67 to 33.32 g U/L.

Wt% uranium would then range from 1.67*100/1,300 = 0.128% or 1,280 μ g/g, to 33.32*100/1,300 = 2.5% or 25,000 μ g/g. These would increase by 25 to 30%, if the original water content of the sludge (~75 wt%) decreased to 50 wt%.

APPENDIX B

STATISTICAL INTERPRETATIONS

This page intentionally left blank.

Westinghouse Hanford Company

Internal Memo

From:

Process Laboratories and Technology

12100-PLT93-070.R1

Phone:

3-4034/3-2779 T6-07/T6-07 August 18, 1993

Date:

Subject: STATISTICAL ANALYSIS OF TANK 241-C-109 DATA

To:	B. C. Simpson	R2-12		
	cc: H. Babad G. L. Borsheim D. A. Dodd J. G. Hill D. A. Reynolds	R2-78 R2-11 T6-50 R2-12 R2-11	J. P. Sloughter R. L. Weiss T. L. Welsh W. I. Winters KMR:LJ File/LB	T6-07 H4-23 T6-07 T6-50

Attached to this revised letter is a report entitled "Statistical Characterization Report for Single-Shell Tank 241-C-109." Format and minor text revisions were made to the original report issued August 13, 1993. The minor text changes are redlined. This report contains a statistical evaluation of the core composite data and the subsegment data from the three core samples. The six topics addressed are as follows:

- Estimates of the mean concentration of analytes found in the single-shell tank and confidence intervals (CI) on the mean
- The results of a statistical test used to determine the Analytical Chemistry Laboratory's (325 Building) ability to homogenize solid core segments
- The results of a statistical test conducted to determine the Analytical Chemistry Laboratory's ability to construct core composite samples from subsegment samples
- Estimates of the spatial variance and the analytical measurement variance and their CIs
- The results of a multiple comparisons procedure applied to the core composite samples and to the subsegment samples
- A comparison of results from tanks 241-C-109 and 241-C-112.

K. M. Remund, Advanced Statistician Process Laboratories and Technology

L. Jensen, Principal Statistician Process Laboratories and Technology

llu

Attachment to 12100-PLT93-070.R1

Statistical Characterization Report for Single-Shell Tank 241-C-109

Page 1 of 52

Statistical Characterization Report for Single-Shell Tank 241-C-109

1.0 SUMMARY

This report contains the results of a statistical analysis of data from three core samples taken from Single-Shell Tank (SST) 241-C-109 (Cl09). Six topics are addressed; they are given in Section 3 through Section 8.

Section 3 contains mean concentration estimates of several analytes found in the SST. In addition, 95% confidence intervals (CI) on the mean concentration are also given.

Section 4 contains the results of a statistical test conducted to determine the Analytical Chemistry Laboratory's (325 Building) ability to homogenize solid core segments. For 16 out of 17 analytes, the variability between sampling locations could not be distinguished from zero. Based upon the results of this statistical test, it is concluded that the Analytical Chemistry Laboratory can satisfactorily homogenize core segments.

Section 5 contains the results of a statistical test conducted to determine the Analytical Chemistry Laboratory's ability to construct core composite samples from subsegment samples. Based upon the large spatial variance and analytical measurement variance, the comparisons showed that the Analytical Chemistry Laboratory's ability to construct core composite samples was satisfactory.

Section 6 contains estimates of the spatial variance (variability between cores) and the analytical measurement variance associated with the core samples. CIs are given for both variances. In 28 out of 33 cases (85%) the spatial variability in the core composite samples was larger than the analytical error.

Section 7 contains the results of a multiple comparisons procedure applied to the means from the core composite samples and to the means from the subsegment samples. There is no significant difference between the composite sample means for 12 of 31 (39%) analytes and they were significantly different in 19 out of 31 (61%) of the analytes. The comparisons between the subsegments indicates "layering" within the waste for some analytes and that the waste is very heterogeneous for other analytes. These types of heterogeneity influence the magnitude of the spatial variability.

Section 8 contains a comparison of results from tanks 241-C-109 and 241-C-112. Due to large spatial and analytical measurement variances and small degrees of freedom, the mean concentrations cannot be distinguished from each other. For most of the analytes, the spatial variances cannot be distinguished from each other. Even though the analytical results came from the same laboratory, the analytical measurement variances were significantly different for 13 out of 28 (46%) analytes.

Appendix 1 contains tables listing the data and the summary statistics. Appendix 2 contains plots of the core composite sample data and subsegment data. Appendix 3 contains plots comparing core composite sample data and subsegment sample data from SSTs 241-C-109 and 241-C-112.

2.0 INTRODUCTION

Three cores samples (cores 47, 48 and 49) were taken from SST C109. Core 47 consisted of three subsegments (denoted by B, C, and D), core 48 consisted of two subsegments (C and D), and core 49 consisted of three subsegments (B, C, and D). The results from a chemical analysis of the cores was used to obtain estimates of the mean concentration of analytes in the waste. In addition, the data was used to evaluate the Analytical Chemistry Laboratory's ability to homogenize subsegments and to construct core composite samples.

Composite samples for each core were made from homogenized subsegment waste. A single composite sample was also made from all of the drainable liquids. Two samples, the sample and the duplicate, were taken from each core composite and subsegment. For the homogenization test, additional samples and duplicate samples were taken from two different locations within the mixed waste.

The laboratory results from SST C109 samples are given in Appendix 1. The analytes of interest from the Inductively Coupled Plasma (ICP) analyses are Al, Ca, Fe, Na, Ni, Pb, U, and P. The ICP acid digestion, and water leach analyses were performed on all composite core samples. The ICP KOH/Ni fusion dissolution analyses were performed on the subsegments and core composite samples. Radio chemistry results on core composite samples were reported for U, Pu-238, Pu-239/240, Cs-137, and Sr-90. A radio chemistry analysis on the subsegments was performed only for Cs-137 and Sr-90. Each subsegment and core composite sample was analyzed by Ion Chromatography (IC) for Chloride, Nitrite, Nitrate, Phosphate, Sulfate and Total Cyanide (CN). In the tables in Appendix 1, the data is identified by the analysis method and the type of dissolution; e.g., the notation ICP.acid.Al refers to aluminum, acid digestion and an ICP analysis.

The core composite sample results are contained in Table 1. Table 2 contains the subsegment sample results. The homogenization test results are contained in Table 3.

Appendix 2 graphically shows the data for core composite and subsegment samples.

A close examination of the data revealed several potential anomalies. These potential anomalies are listed below.

The following core composite sample results were an order of magnitude different (lower or higher) than other core composite sample results:

- core 48 ICP.acid.Al
- core 47 ICP.acid.Pb
- core 48 ICP.fusion.Al

- core 49 ICP.fusion.Pb
- core 47 Total Alpha Pu
- core 47 Pu-239/240.

Core 47 subsegment 1B result for ICP.fusion.Fe is at least twice as large as its duplicate and all other subsegment data for this analyte.

The following subsegments were different by an order of magnitude (lower or higher) than other subsegment results:

- core 48 subsegments 1C and 1D for ICP.fus.AT
- core 47 and 48 subsegment 1B for fusion.Sr-90.

There is no direct evidence that the results noted above are due to analytical measurement errors. Consequently, the statistical analysis was performed on the data as it is reported in Tables 1, 2, and 3.

3.0 MEAN CONCENTRATION ESTIMATES

A task outlined in the Waste Characterization Plan was to estimate the analyte concentrations in the waste (Winters, 1990). They were estimated by computing mean concentrations and 95% CIs on the mean concentrations. The estimate of the inventory and CI on the inventory of an analyte, are the corresponding mean concentration estimates and CI multiplied by the volume of waste in the tank. These estimates are not given in this document.

Table 1 contains the core composite data used to compute the mean concentration estimates and the CIs. The "NA" symbol indicates that the data was not available. Results for Pu-238 were not included in any computations since there were no duplicate measurements.

3.1 Statistical Methods

The concentration estimates are given in the form of 95% CIs on the mean concentration. It is assumed that each sample and duplicate sample are analyzed independently of one another. The two analytical results are used to estimate the analytical measurement error. Due to the hierarchical structure of the data, the analytical measurement error (variance) alone is not the appropriate error term to use in computing the CIs. A linear combination of the analytical measurement variance and spatial variance is the appropriate variance of the mean for the CIs. The variance of the mean is obtained from the analysis of variance (ANOVA) corresponding to the model. Brown (1993), Appendix G, gives the formula used to calculate these CIs.

3.2 Statistical Results

Table 4 contains the summary statistics, by analyte, for ICP acid digestion, ICP water leach, ICP KOH\Ni fusion dissolution, radio chemistry, and IC

analyses. Table 5 contains the summary statistics for the drainable liquid composite sample. The summary statistics are as follows:

$\hat{\sigma}^2(\bar{y})$ estimated variance of \bar{y}

df degrees of freedom associated with BMS

95% LL lower limit to the 95% CI on the mean

95% UL upper limit to the 95% CI on the mean.

For some analytes the lower confidence limit (95% LL) was negative. Since concentrations are strictly greater than or equal to zero, any negative 95% LL values were set equal to zero.

The estimated variance of the mean $(\hat{\sigma}^2(\bar{y}))$ is very large relative to the mean for most of the analytes. One cause is the large differences between core composite samples; i.e., the large spatial variability. In Section 7 on multiple comparisons, the analytical results from the core composite samples are compared to determine if there are significant differences between cores. A similar comparison is also made between the subsegments within each core.

4.0 HOMOGENIZATION TEST

A second task in applying the Waste Characterization Plan was to evaluate the ability of the Analytical Chemistry Laboratory to homogenize subsegments (Winters, 1990).

Subsegment D, from cores 47, 48, and 49, was homogenized and arbitrarily divided into two parts. One subsample was obtained from each part. Two aliquots were taken from each subsample and prepared for chemical analysis. The homogenization test data is given in Table 3. ICP acid digestion and fusion dissolution analyses were conducted on the samples for the following analytes: Al, Ca, Fe, Na, Ni, Pb, U, and P. Acid digestion and fusion dissolution results were also reported for Cs-137.

4.1 Statistical Methods and Results

Due to the nested structure (subsamples within subsegments, aliquots within subsamples) within the data, a hierarchical statistical model was fit to the data. Snedecor (1980), page 284, contains a description of this type of model. Such a model is used to estimate different components of variability in the data. The total variability in the data is decomposed into three components: one due to variability between subsegments, one due to the variability between samples taken from different locations on each homogenized subsegment ($\sigma^2(L)$), and one due to the analytical measurement error ($\sigma^2(A)$). The analytical measurement error accounts for the differences between aliquots taken from the same location.

To quantify the contribution of $\sigma^2(L)$ (the component of variability due to location or homogenization), the ANOVA corresponding to the hierarchical model is used. From the ANOVA, a test is constructed to determine if $\sigma^2(L)$ is significantly different from zero. If $\sigma^2(L)$ is significantly different from zero, then the laboratory does not have the ability to homogenize subsegments. If $\sigma^2(L)$ is not significantly different from zero, then the laboratory has the ability to homogenize core segments. The reason underlying this test is that if $\sigma^2(L)=0$, then the mean concentrations at the two locations are equal; i.e., there is no difference between the locations.

The F-test is used to determine whether or not $\sigma^2(L)$ is significantly different from zero. The p-values (the attained level of significance) from these tests are given in Table 6. If the p-value is smaller than 0.05, then $\sigma^2(L)$ is significantly different from zero. In all but one case (ICP.fus.Al), the p-values are greater than 0.05. This indicates that, except for this analyte, $\sigma^2(L)$ is not significantly different from zero. Based upon the results of this statistical test it can be concluded that the Analytical Chemistry Laboratory can adequately homogenize core segments. However, it needs to be noted that there is no reference value available to check the degree of homogenization; e.g., the differences between the results from the two locations must be within 2% of each other. If such a value were available, the conclusions in this section may not be valid.

5.0 COMPARISON WITH A SIMULATED CORE COMPOSITE MEAN

Another task in applying the Waste Characterization Plan was to evaluate the ability of the Analytical Chemistry Laboratory to make core composite samples from the individual subsegment samples (Winters, 1990). Core composite samples were formed by combining samples from each homogenized subsegment in the core. Each subsegment is weighted equally in the composite sample. Each core composite sample was homogenized, and a sample and duplicate sample obtained.

A simulated core composite (SCC) was statistically constructed to compare to the corresponding core composite sample results. For each analyte and each core, the SCCs are the average of the subsegment results. This mean or average is denoted by $\overline{y}(w)$. The "w" is used since $\overline{y}(w)$ is generally a weighted mean. However, in this case the weights are all equal.

5.1 Statistical Methods

For each core, the comparison between the core composite and SCC is made by computing a CI on the difference between the SCC and the mean of the composite sample. If zero is in the CI, then the laboratory can construct core composite samples satisfactorily (i.e., the SCC cannot be statistically distinguished from the core composite sample mean). If zero is not in the CI, then the laboratory cannot satisfactorily construct core composites (i.e., the two means are significantly different).

The CI for this difference is (LL, UL) where the lower limit (LL) and upper limit (UL) values are

$$LL = [\overline{y}(w) - \overline{y}(c)] - t\sqrt{\hat{\sigma}^2[\overline{y}(w) - \overline{y}(c)]}, \quad UL = [\overline{y}(w) - \overline{y}(c)] + t\sqrt{\hat{\sigma}^2[\overline{y}(w) - \overline{y}(c)]}$$

where

y(c) mean of the two core composite sample results,

t percentile point from Student's t distribution, and

 $\hat{\sigma}^2[\bar{y}(w)-\bar{y}(c)]$ is the estimated variance of the difference.

Appendix 4 outlines the method used to calculate $\hat{\sigma}^2[\bar{y}(w)-\bar{y}(c)]$. The estimated variance $\hat{\sigma}^2[\bar{y}(w)-\bar{y}(c)]$ was calculated using the data from all three cores because of the limited information available. The degrees of freedom (df) associated with "t" were calculated using Satterthwaite's approximation (Snedecor, 1980, page 228). In the above equations, $\bar{y}(w)$ and $\bar{y}(c)$ should have a subscript indicating the core. To simplify the notation, the subscript is omitted.

5.2 Statistical Results

Table 7 contains summary statistics for all three cores, including the 95% CI interval (LL,UL) on the difference between the SCC mean and the core composite mean. All of the CIs on this difference contain zero. This indicates that there is no significant difference between the two means (i.e., the Analytical Chemistry Laboratory can construct a core composite sample from material similar to cores 47, 48, and 49).

It needs to be pointed out that (see Table 7) the CIs on the difference between the two means tend to be rather wide; i.e., $LL=-10^5$ or -10^4 and $UL=+10^5$ or $+10^4$. The reason for this extreme width is the magnitude of the estimated variance $\hat{\sigma}^2[\bar{y}(w)-\bar{y}(c)]$ and the small number of degrees of freedom. Since the variance is large, the two means would have to be extremely different before zero in not in the CI.

6.0 THE SPATIAL VARIANCE AND ANALYTICAL MEASUREMENT VARIANCE

Using the hierarchical structure of the core composite data, the spatial variance and the analytical measurement variance can be separated from each other. The spatial variance is a measure of the variability between cores. The analytical measurement variance includes, among other things, the segment homogenization error, the sample handling error, and the chemical analysis error. This variance is a function of the difference between the analytical results on the sample and duplicate sample.

The size of the analytical measurement variance and the spatial variance, along with the degrees of freedom, determine the width of the CIs. The

estimate of the variance of the mean is a linear function of the spatial and analytical measurement variances. To help judge the magnitude of these two variance components, this section contains explicit estimates of each variance and CIs for each variance.

6.1 Statistical Methods

Estimates of the spatial variance $(\hat{\sigma}^2(S))$ and analytical measurement variance $(\hat{\sigma}^2(A))$ were obtained for each analyte using Restricted Maximum Likelihood Estimation. This method is discussed by Harville (1977). Snedecor (1980), page 246, outlines methods that can be used to obtain CIs for $\hat{\sigma}^2(S)$ and $\hat{\sigma}^2(A)$. These CI techniques are the methods used in this document. The CI for $\hat{\sigma}^2(S)$ is approximate. The CI for $\hat{\sigma}^2(A)$ is exact.

6.2 Statistical Results

Tables 8 and 9 contain estimates of the variance components and their 95% CIs. For 85% of the analytes (28 out of 33), the estimates of spatial variance are larger than those for the analytical error. This large spatial variability contributes to the extreme width of the CI for the mean concentrations, and the CI on the difference between the synthetic core composite and the core composite sample.

Section 8 contains estimates of relative standard deviations of the mean, the analytical measurement variance, and the spatial variance. In addition, Section 8 also contains estimates of the contribution of $\hat{\sigma}^2(A)$ and $\hat{\sigma}^2(S)$ to the total variance.

7.0 MULTIPLE COMPARISONS: CORE COMPOSITE SAMPLES AND SUBSEGMENT SAMPLES

A group of statistical methods known as multiple comparisons can be used to determine whether or not there are significant differences between core composite samples and between subsegment samples. These differences will aid in determining heterogeneity or layers within the waste. In addition, if significant differences exist between the core composite samples or the subsegment samples, then this will help explain the extreme width of the CIs; i.e., it will help explain the large spatial variability.

7.1 Statistical Methods

The multiple comparison procedure known as Tukey's Honestly Significant Difference (HSD) (Petersen, 1985, page 78) was used. The HSD procedure determines if there are significant differences between core composite samples and between subsegment samples. The core composite samples and subsegment samples that are not significantly different from each other can then be grouped together.

7.2 Statistical Results

For each analyte, HSD comparisons were made between the means of the core composite samples. These comparisons, along with the means for each core

composite sample, are contained in Table 10. The symbols α and β are used to indicate groupings. The means of core composite samples with the same symbol cannot be statistically distinguished from each other. Core composite samples with different symbols are significantly different from each other. For a given analyte, the core composite samples with an α have a smaller mean concentration than the core composite samples with a β . A dash indicates that no data was available. The HSD comparisons are not based upon the spatial variance; they are however, a function of the analytical measurement variance.

From Table 10, it is evident that the mean concentration of 12 out of 31 (39%) analytes are not significantly different between the cores. That is, for these analytes there is no evidence of heterogeneity within the waste. For the remaining 19 (61%) analytes there is a significant difference between the mean concentrations. This indicates significant heterogeneity or spatial variability within the waste. These significant differences inflate the between core variance (spatial variance). This inflated variance helps explain why the CIs are so wide.

Examples of the interpretation of the HSD comparisons are as follows. In Table 10, Al (ICP.acid) has an α for core 48 and a ß for core 47 and 49. That is, the mean concentration of Al in core 48 is significantly different from the mean concentration in core 47 and 49. The mean concentration of Al in core 47 and 49 cannot be distinguished from each other. Another example is U (ICP.acid). In Table 10, core 48 has a ß, core 49 has an α , and core 47 has an $\alpha\beta$. That is, the mean concentration of U for core 48 is significantly different from the mean concentration in core 49. Core 47 has both symbols α and β . So that the mean concentration of U is not significantly different from the mean concentration in core 48 and it is not significantly different from the mean concentration in core 49.

There were three subsegment samples from cores 47 and 49 (denoted by B, C, and D) and two from core 48 (denoted by C and D). The relative location of the subsegments are given in the following table.

Core	47	48	49
Subsegment	B C	С	B C
	D	_ D _	D

Tukey's HSD procedure was also used to make comparisons between the individual subsegment means. These comparisons, along with subsegment means, are given in Table 11. For a given analyte, the relative locations of the subsegments have the form given in the above table. The symbols used to denote groupings of means concentrations are α , β , γ , δ , ϵ , and ϕ . A dash indicates no data was available. The interpretation and ranking of the groupings is identical to that given for Table 10.

The results given in Table 11 appear to be complicated. A partial explanation is as follows. The multiple comparisons indicate that the three subsegments between cores 47 and 49 match for Na, Pb, Chloride, Nitrate, Phosphate, and Cs-137. Subsegments B and D match between cores 47 and 49 for Fe, Nitrite, Sulphate and Total CN. Subsegments C and D match between cores 47, 48, and 49 for Fe and Sr-90. This is some evidence of "layers" in the waste. The multiple comparisons for the other analytes indicate waste heterogeneity. As was stated above, such differences inflate the spatial variance. There may be patterns in Table 11, other than those indicated, showing "layers" within the waste.

8.0 COMPARISON OF RESULTS: SST 241-C-109 and 241-C-112

This section contains a comparison of summary statistics computed from core composite data from tanks ClO9 and Cll2. The comparison is made on the mean concentration, the analytical measurement variance, and the spatial variance. Comparisons are also presented for the relative standard deviations and the two variances as a percent of the total variance.

Appendix 3 contains graphs of the data from C109 and C112. Each core from C109 is paired, according to location within the tank, with a core from C112. The following table shows this pairing:

Tank	Pair 1	Pair 2	Pair 3
C109	Core 49	Core 48	Core 47
C112	Core 34	Core 35	Core 36

To aide in the visual comparisons between the two sets of data, paired cores have the same symbols.

8.1 Comparison of Mean Concentrations

For the analytes of interest in tank Cl09, the mean concentration (\bar{y}) and the variance of the mean concentration $(\hat{\sigma}^2(\bar{y}))$ are presented in Table 4. These summary statistics are based upon the core composite data. Tables 7 to 11 of Appendix B, Simpson (1993), contain the corresponding results for tank Cl12.

To test the equality of the mean concentration of the analytes in the two tanks, a 95% CI on the difference between the two mean concentrations was computed. The test of equality of mean concentrations is: if zero is in the CI, then the two means cannot be distinguished from each other at the 0.05 level of significance. If zero is not in the CI, the two means are significantly different from each other.

A 95% CI on the difference between the mean concentrations was computed for 29 analytes. The CI was based on Cochran's approximation to the Behrens-Fisher problem (Snedecor, 1980, page 97). Zero was in all of the intervals. Due to

the magnitude of the variances of the difference, $\hat{\sigma}^2[\bar{y}(\text{Cl09})-\bar{y}(\text{Cl12})]=\hat{\sigma}^2[\bar{y}(\text{Cl09})]+\hat{\sigma}^2[\bar{y}(\text{Cl12})]$ and the small number of degrees of freedom, the width of the CIs was very large. The lower limits of the intervals tended to be -10^5 or -10^4 and the upper limits tended to be $+10^5$ or $+10^4$. These intervals are a function of both the analytical measurement variance and the spatial variance.

The CIs were also computed using only the analytical measurement variance. The new lower limits of the intervals were reduced to -10^4 or -10^3 and the upper limits reduced to $+10^4$ or $+10^3$. Zero was in all but two of the new CIs. The two intervals that did not contain zero were for Al (ICP.fusion) and Pu-239/240. Tables containing the CIs on the difference are not given in this document.

The interpretation of these results are that, except for possibly Al (ICP.fusion) and Pu-239/240, the mean concentration of the analytes in the two tanks cannot be distinguished from each other. It needs to be emphasized that these comparisons are based upon very large spatial and analytical measurement variances and very small degrees of freedom. Consequently, such comparisons may not be meaningful.

8.2 Comparison of Variances

The estimates of the analytical measurement variances $(\hat{\sigma}^2(A))$ for both C109 and C112 are given in Table 12. A special form of the F-test, (Snedecor, 1980, page 98) was used to test the equality of these two variances. The p-value, which is the attained level of significance of the F-test, is also given in Table 12. If the attained level of significance is less than 0.025 (in this special test), then the two analytical measurement variances are significantly different from each other at the 0.05 level of significance. In 13 out of 28 tests (46%) the analytical measurement variances were significantly different from each other for the two tanks.

The equality of the spatial variances for the two tanks was tested in a similar manner. Table 13 contains the estimates of the spatial variances $(\hat{\sigma}^2(S))$ and the p-values for the F-test. The spatial variances were significantly different from each other in only 4 out of 25 tests (16%). These variances were significantly different for U (ICP.acid), for Ni (ICP.water) and for U and Pu-239/240. The spatial variances cannot be distinguished from each other for the other analytes.

The implication of these results are that, at least for these two tanks, the degree of heterogeneity in the waste is very similar. The analytical measurement error is not consistent between the two tanks, even though the data was analyzed by the same laboratory. These results must be interpreted with caution, since there are only two degrees of freedom associated with each spatial variance and two or three degrees of freedom for each analytical measurement variance. The degrees of freedom are very small.

Results given in Tables 14 and 15 may help in the interpretation of the relative magnitude of the estimates of the analytical and spatial variances. Table 14 gives, for both tanks Cl09 and Cl12, $\hat{\sigma}^2(A)$ and $\hat{\sigma}^2(S)$ as a percent of

Ī ...

the total variance associated with any observation. That is, $\hat{\sigma}^2(A) = 100 * \hat{\sigma}^2(A) / [\hat{\sigma}^2(A) + \hat{\sigma}^2(s)]$ and $\hat{\sigma}^2(S) = 100 * \hat{\sigma}^2(S) / [\hat{\sigma}^2(A) + \hat{\sigma}^2(s)]$. Generally, $\hat{\sigma}^2(s)$ represents the greatest percentage of the total variability for both C112 and C109.

The relative standard deviations (RSDs) for both tanks are given in Table 15. An RSD is a standard deviation expressed as a percent of the mean concentration. That is, RSD(\bar{y})=100* $\hat{\sigma}(\bar{y})/\bar{y}$, RSD(A)=100* $\hat{\sigma}(A)/\bar{y}$, and RSD(S)=100* $\hat{\sigma}(S)/\bar{y}$. The RSD(A) appears to be relatively consistent between the two tanks. The RSD(S) appears to be variable with no apparent pattern.

9.0 REFERENCES

- Brown, T. M., and L. Jensen, 1993, *Tank Characterization Report for Single-Shell Tank 241-U-110*, WHC-EP-0643, Rev. O, Westinghouse Hanford Company, Richland, Washington.
- Harville, D. A., 1977, "Maximum Likelihood Approaches to Variance Component Estimation and to Related Problems," *Journal of the American Statistical Association*, pp. 320-340.
- Petersen, R. G., 1985, Design and Analysis of Experiments, First Edition, Marcel Dekker, Inc., New York.
- Simpson, B. C., et al., 1993, *Tank Characterization Data Report: 241-C-112*, WHC-EP-0640, Westinghouse Hanford Company, Richland, Washington.
- Snedecor, G. W. and Cochran, W. G., 1980, Statistical Methods, Seventh Edition, Iowa State University Press, Ames, Iowa.
- Winters, W. I., et al., 1990, Waste Characterization Plan for the Hanford Site Single-Shell Tanks, WHC-EP-0210, Rev. 1, Westinghouse Hanford Company, Richland, Washington.

APPENDIX 1: TABLES OF DATA AND STATISTICAL RESULTS

Table 1. Core Composite Data (Units $\mu g/g$ Except Radionuclides $\mu Ci/g$).

Core	4	7	_ 4	8	49	
Analysis	.1	2	1	2	1	2
ICP.acid.Al	7.41e+04	7.16e+04	6.24e+03	6.60 e+ 03	9.59e+04	7.15e+04
ICP.acid.Ca	1.95e+04	2.05e+04	1.44e+04	1.07e+04	1.38e+04	1.08e+04
ICP.acid.Fe	3.52+04	2.24+04	1.39e+04	2.65+04	8.39 e+ 03	5.90e+03
ICP.acid.Na	8.15e+04	8.22e+04	8.16e+04	9.35e+04	6.58e+04	9.68e+04
ICP.acid.Ni	1.46e+04	1.49e+04	1.63e+04	1.47e+04	1.31e+04	1.0 6e+ 04
ICP.acid.Pb	9.96e+03	7.25e+03	5.86e+02	6.2 6e+ 02	9.99e+02	7.28 e+ 02
ICP.acid.U	1.05e+04	1.10e+04	1.27e+04	1.74e+04	7.10e+03	5.43e+03
ICP.acid.P	1.84e+04	1,84e+04	1.45e+04	1.96e+04	1.17e+04	2.71e+04
ICP.fusion.Al	1.15e+05	1.19e+05	7.28e+03	9.86e+03	1.20e+05	1.34e+05
ICP.fusion.Ca	2.44e+04	2.49+04	1.68e+04	1.85e+04	1.45e+04	1.52e+04
ICP, fusion.Fe	2.02e+04	2.34e+04	2.38e+04	2.06e+04	9.27e+03	8.94 e+ 03
ICP.fusion.Na	8.71e+04	8.72e+04	1.07e+05	9.33e+04	8.18e+04	7.13e+04
ICP.fusion.Pb	7.22e+03	7.34e+03	NA NA	NA	8.03e+02	8.44e+02
ICP.fusion.U	8.75e+03	9.61e+03	2.78e+04	2.17e+04	5.59e+03	NA
ICP.fusion.P	2.02e+04	1.96e+04	2.22e+04	1.82 e+ 04	1.77e+04	1.14e+04
ICP.weter.Al	3.36e+02	4.88e+02	NA.	NA	NA	NA
ICP.water.Ca	1.73e+02	1.94e+02	5.93e+01	5.97e+01	8.92e+01	6.62e+01
ICP.weter.Fe	8.85e+02	8.72e+02	1.13e+03	1.15e+03	8.88e+02	9.44e+02
ICP.water.Ne	6.60e+04	6.96e+04	8.92e+04	7.79e+04	5.89e+04	6.09 e+ 04
ICP.water.Ni	1.40e+02	1.09e+02	3.34e+01	2.85e+01	5.28e+01	5.28e+01
ICP.water.P	6.35e+03	7.63#+03	1.19 e+ 04	5.46e+03	4.42e+03	3.90 e+ 03
Chloride	7.00e+02	7.00e+02	8.00e+02	8,00e+02	7.00e+02	7.00e+02
Nitrite	3.80e+04	4.00e+04	4.20e+04	4.80e+04	3.80e+04	3.90e+04
Nitrate	3.70e+04	3.70e+04	4.50e+04	5.10e+04	3.50e+04	3.70e+04
Phosphate	2.01e+04	2.40e+04	3,59e+04	1.75e+04	1.35e+04	1.20e+04
Sulfate	7.20e+03	8.10e+02	8.90e+03	9.60e+03	6.20e+03	6.90e+03
Total CN	5.60e+03	5.41e+03	1.41e+04	1.46e+04	5.64e+03	5.59e+03
U (µg/g)	1.17e+01	1.22e+01	3.00e+01	2.51e+01	7.63e+00	7.42e+00
Total Alpha (Pu)	8.05e-01	9.49e-01	6.95e-02	6.66e-02	6.59e-02	9.21e-02
Sr-90	1.05e+03	1.30e+03	1.90e+02	1.90e+02	8.77e+02	9.86e+02
Pu- 238	4.40e-05	NA	7.15e-06	NA	1.11e-05	NA NA
Pu-239/240	8.04e-01	9.48e-01	6.95e-02	6.66e-02	6.58e-02	9.20e-02
Cs-137/water	9.07e+00	9.40e+00	7.95e+00	1.07e+01	5.61e+00	4.95e+00
Cs-137/fusion	8.70e+02	8.77e+02	1.11e+03	9.52e+02	5.47e+02	5.66e+02

Table 2. Subsegment Data (Units $\mu g/g$ Except Radionuclides $\mu Ci/g$).

Core	}	47		4	8		49	
Subsegment	В	С	D	С	D	В	С	D
ICP.fusion.Al	1.24e+05	1.20e+05	3.27e+04	7.44e+03	9.60e+03	1.81e+05	9.75e+04	7.35e+04
	1.39e+05	1.21e+05	3.13e+04	7.14e+03	1.01e+04	1.90e+05	9.40e+04	6.82e+04
ICP.fusion.Ca	1.07e+04	1.84e+04	2.88e+04	3.02e+04	1.70e+04	5.40e+03	1.89e+04	2.13e+04
	1.02e+04	1.77e+04	2.72e+04	2.84e+04	1.66e+04	3.21e+03	1.82e+04	2.37e+04
ICP.fusion.Fe	8.25e+04	1.54e+04	1.71e+04	2.28e+04	2.27e+04	1.38e+04	4.33e+03	1.36e+04
	4.42e+04	2.65e+04	1.35e+04	1.72e+04	1.94e+04	1.74e+04	4.82e+03	1.72e+04
ICP.fusion.Na	4.97e+04	6.32e+04	1.02e+05	1.38e+05	1.01e+05	4.51e+04	6.09e+04	9.02e+04
	5.24e+04	6.29e+04	1.04e+05	9.33e+04	1.03e+05	4.10e+04	6.48e+04	9.25e+04
ICP.fusion.Pb	5.53e+03	2.99e+03	1.86e+04	NA	7.24e+02	2.07e+03	AA	6.95e+02
	4.57e+03	2.78e+03	1.00e+04	NA	6.62e+02	1.90e+03	NA	7.62e+02
ICP.fusion.U	1.15e+04	6.61e+03	6.24e+03	1.81e+04	1.46e+04	8.66e+03	NA	1.15e+04
	1.20e+04	5.68e+03	5.44e+03	1.54e+04	1.41e+04	7.15e+03	NA	1.32e+04
ICP.fusion.P	NA	1.26e+04	2.90e+04	2.62e+04	2.02e+04	4.57e+03	1.14e+04	2.05e+04
	7-89e+03	1.23e+04	3.12e+04	2.03e+04	2.16e+04	NA	1.16e+04	2.03e+04
Chloride	5.00e+02	7.00e+02	8.00 e+ 02	1.00e+03	1.00e+03	5.00e+02	8.00e+02	8.00e+02
	6.00e+02	7.00e+02	7.00e+02	9.00e+02	1.00e+03	5.00e+02	8.00e+02	8.00e+02
Nitrite	2.70e+04	3.70e+04	4.00e+04	4.90e+04	4.90e+04	2.58e+04	4.20e+04	4.60e+04
	2.88e+04	3.70e+04	3.90e+04	5.30e+04	5.00e+04	2.71e+04	4.50e+04	4.40e+04
Nitrate	2.69e+04	3.60e+04	3.90e+04	5.50e+04	5,20e+04	2.52e+04	4.00e+04	4.40e+04
	2.83e+04	3.60e+04	3.80e+04	5.70e+04	5,50e+04	2.62e+04	4.40e+04	4.20e+04
Phosphate	7.10e+03	9.60e+03	3.40e+04	1.50e+04	3.80e+04	6.00e+03	8.90e+03	2.43e+04
	7.50e+03	9.50e+03	5.50e+04	1.65e+04	3.40e+04	6.20e+03	8.70e+03	2.60e+04
Sulfate	4.90e+03	7.10e+03	7.60e+03	1.08e+04	1.00e+04	4.50e+03	7.90e+03	7.90e+03
	5.20e+03	7.10e+03	7.10e+03	1.12e+04	1.00e+04	4.80e+03	8.40e+03	8.30e+03
Total CN	3.05e+03	4.49e+03	5.83e+03	1.10e+04	8.60e+03	3.50e+03	8.14e+03	5.61e+03
	3.03e+03	4.23e+03	5.82e+03	1.15e+04	8.71e+03	3.57e+03	8.02e+03	5.43e+03
Sr-90-fusion	4.60e+03	4.56e+02	2.31e+02	1.59e+02	1.27e+02	2.56e+03	2.02e+02	1.88e+02
	4.51e+03	4.82e+02	1.99e+02	1.44e+02	1.14e+02	2.23e+03	1.89e+02	1.97e+02
Cs-137. fusion	3.17e+02	8.12e+02	9.71e+02	1.17e+03	1.22e+03	1.21e+02	5.53e+02	6.60e+02
	3.57e+02	7.31e+02	9.23e+02	1.14e+03	1.11e+03	1.15e+02	1.44e+02	7.43e+02

Table 3. Homogenization Test Data (Units $\mu g/g$ Except Radionuclides $\mu Ci/g$).

Core		18	49			
Subsegment	D	D	D	D		
Location	1	2	1	2		
ICP.acid.Al	8.73e+03	9.45 e+ 03	3.94e+04	4.18e+04		
	7.89e+03	8.84 e+ 03	4.66e+04	4.68e+04		
ICP.acid.Ca	1.53e+04	1.73e+04	1.46e+04	1.6 9e+ 04		
	1.42e+04	1.56e+04	1.94e+04	1.87 e+ 04		
ICP.acid.Fe	2:38e+04	1.78e+04	8.85e+03	9.05e+03		
	1:37e+04	1.68e+04	1.15e+04	1.12e+04		
ICP.acid.Na	1.16e+05	9.93e+04	1.16e+05	8.80e+04		
	1.21e+05	1.06e+05	8.66e+04	8.01e+04		
ICP.acid.Ni	1.71e+04	1.94e+04	1.19e+04	1.29e+04		
	1.56e+04	1.74e+04	1.56e+04	1.48e+04		
ICP.acid.Pb	6.17e+02	7.23e+02	4.85e+02	5.08e+02		
	5.68e+02	6.45e+02	6.70e+02	6.16e+02		
ICP.acid.U	1.54e+04	1.74e+04	9.71e+03	1.00e+04		
	1.45e+04	1.55e+04	1.34e+04	1.21e+04		
ICP.acid.P	2.69e+04	1.96e+04	3.23e+04	2.09e+04		
	3.08e+04	2.29e+04	1.86e+04	1.75e+04		
ICP.fusion.Al	NA	NA	6.17e+04	5.31e+04		
	NA	NA	6.30e+04	5.59e+04		
ICP.fusion.Ca	NA	NA	2.17e+04	2.14e+04		
	NA	NA	2.22e+04	2.17e+04		
ICP.fusion.Fe	NA	NA	1.37e+04	1.28e+04		
	NA	NA	1.44e+04	1.28e+04		
ICP.fusion.Na	NA	NA	9.08e+04	9.02e+04		
	NA	NA	9.05e+04	8.92e+04		
ICP.fusion.Ni	NA	NA	N/A	N/A		
	NA	NA	N/A	N/A		
ICP.fusion-Pb	NA	NA	6.25e+02	6.46e+02		
	NA	NA	7.45e+02	6.50e+02		
ICP.fusion.U	NA	NA	1.23e+04	1.08e+04		
	NA	NA	1.31e+04	1.18e+04		
ICP.fusion.P	NA	NA	1.87e+04	1.95e+04		
	NA	NA	1.91e+04	1.86e+04		
Cs-137.fusion	NA	NA	7.13e+02	6.96e+02		
	NA	NA	7.50e+02	7.00e+02		
Cs-137.acid	8.52e+00	8.81e+00	3.54e+01	1.93e+01		
	1.66e+01	1.43e+01	4.34e+01	2.74e+01		

Table 4. Concentration Estimate Statistics (Units μ g/g Except Radionuclides μ Ci/g).

Analyte	ÿ	∂ ² (ÿ)	df	95% LL	95% UL
ICP.acid.Al	5.43e+04	5.84e+08	2	0.00e+00	1.58e+05
ICP.acid.Ca	1.50e+04	6.38e+06	2	4.10e+03	2.58e+04
ICP.acid.Fe	1.87e+04	3.96e+07	2	0.00e+00	4.58e+04
ICP.acid.Na	8.36e+04	4.02e+06	2	7.49e+04	9.22e+04
ICP.acid.Ni	1-40e+04	1.23e+06	2	9.27e+03	1.88e+04
ICP.acid.Pb	3.36e+03	6.89e+06	2	0.00e+00	1.47e+04
ICP.acid.U	1.07e+04	6.40e+06	2	0.00e+00	2.16e+04
ICP.acid.P	1.83e+04	4.73e+05	2 2 2 2 2 2 2	1.53e+04	2.13e+04
ICP_fus_At	8.40e+04	1,43e+09	2	0.00e+00	2.47e+05
ICP.fus.Ca	1.91e+04	8.50e+06	1 5	6.52e+03	3.16e+04
ICP.fus.Fe	1.77e+04	1.85e+07	2 2 2 1	0.00e+00	3.62e+04
ICP.fus.Na	8.79e+04	4.64e+07	1 5	5.86e+04	1.17e+05
ICP.fus.Pb	4.05e+03	1.04+07	l i	0.00e+00	4.51e+04
ICP.fus.U	1.47+04	3.72e+07		0.00e+00	4.09e+04
ICP.fus.P	1.82e+04	3.29e+06	2 2	1.04e+04	2.60e+04
ICP.water.Ca	1.07e+02	1.49e+03	2	0.00e+00	2.73e+02
ICP.water.Fe	9.78e+02	6.61e+03	2	6.28e+02	1.33e+03
ICP .water .Ne	7.04+04	4.82e+07	2	4.05e+04	1.00+05
ICP.water.Ni	6.94e+01	7.98e+02	2	0.00e+00	1.91e+02
ICP.water.P	6.61e+03	1.74e+06	5 5	9.31e+02	1.23e+04
Chloride	7.33e+02	1.11e+03	2	5.90e+02	8.77e+02
Nitrite	4.08e+04	4.36e+06	1 3	3.18e+04	4.98e+04
Nitrate	4.03e+04	1.48e+07	2 2 2 2 2 2	2.38e+04	5-69e+04
Phosphate	2.05e+04	1.68e+07	1 3	2.85e+03	3.81e+04
Sulphate	7.70e+03	6.48e+05	1 5	4.24e+03	1.12e+04
Total CN	8.46e+03	8.39e+06	1 5	0.00e+00	2.09e+04
TOTAL CH			 		
U (μg/g)	1.57e+01	3.69e+01	2	0.00e+00	4.18e+01
Tot.Alpha.Pu	3.41e-01	7.17e-02	2 2	0.00e+00	1.49e+00
Sr-90 °	7.66e+02	8.77e+04] 2	0.00e+00	2.04e+03
Pu-239/240	3.41e-01	7.16e-02	2	0.00e+00	1.49e+00
Cs-137.water	7.95e+00	1.78e+00	2 2 2	2.21e+00	1.37e+01
Cs-137, fusion	8.20e+02	1.95e+04	2	2.20e+02	1.42e+03

Table 5. Concentration Estimates Statistics, Drainable Liquid, (Units $\mu g/g$).

Analyte	ÿ	$\partial^2(\bar{y})$	df	95% LL	95% UL
ICP.acid.Al	1.57e+02	1.50e+01	1	1.08e+02	2.06e+02
ICP.acid.Ca	2.09e+02	1.74e+01	1 1	1.56e+02	2.62e+02
ICP.acid.Fe	1.67e+03	5.56e+02	1 1	1.38e+03	1.97e+03
ICP.acid.Na	9.69e+04	2.66e+01	1 1	9.69e+04	9.70e+04
ICP.acid.Ni	3.44e+02	1.03e+01	1 1	3.03e+02	3.84e+02
ICP.acid.Pb	NA.	NA	NA.	0.00	0.00
ICP.acid.U	NA	NA.	NA.	0.00	0.00
ICP.acid.P	4,20e+03	9.65e+02	11	3.80e+03	4.59e+03
Chloride	1.30e+03	NA NA	NA	NA	NA
Nitrite	7.10e+04	l NA	NA.	NA NA	NA NA
Nitrate	7.20e+04	NA	NA.	NA NA	NA
Phosphate	1.35e+04	NA	NA.	NA	NA
Sulphate	1.28e+04	NA NA	NA	NA NA	NA.

Table 6. Homogenization Test Statistical Results.

Analyte	Test: $\sigma^2(L)*0$ p-value
ICP.acid.Al	0.890
ICP.acid.Ca	0.649
ICP.acid.Fe	0.922
ICP.acid.Na	0.229
ICP.acid.Ni	0.551
ICP.acid.Pb	0.572
ICP.acid.U	0.667
ICP.acid.P	0.290
1CP.fus.Al	0.036
ICP.fus.Ca	0.389
ICP.fus.Fe	0.072
ICP.fus.Na	0.216
ICP.fus.Pb	0.606
ICP.fus.U	0.164
ICP.fus.P	0.706
Cs-137.fus	0.214
Cs-137.acid	0.092

Table 7. Comparison of Simulated Core Composite with the Core Composite.

Core	Analyte	ÿ(¥)	ÿ(c)	δ ² [ỹ(w)-ỹ(c)]	df	95% LL	95% UL
47	ICP.fus.Al	9.46e+04	1.17e+05	5.79e+09	3	-2.64e+05	2.20e+05
l - '	ICP.fus.Ca	1.88e+04	2.47e+04	4.75e+07	6	-2.27e+04	1.10e+04
	ICP.fus.Fe	3.32e+04	2.18e+04	1.60e+08	1 8	-1.77e+04	4.05e+04
ŀ	ICP.fus.Na	7.23e+04	8.72e+04	4.08e+08	7	-6.27e+04	3.29e+04
1 .	ICP.fus.Pb	7.41e+03	7.28e+03	3.00e+07	ĺź	-2.34e+04	2.37e+04
1 1	ICP.fus.U	7.92e+03	9.04e+03	1.15e+08	2	-4.72e+04	4.50e+04
1	ICP.fus.P	1.68e+04	1.99e+04	3.41e+07	9	-1.63e+04	1.02e+04
	Chloride	6.67e+02	7.00e+02	1.50e+04	[š	-3.48e+02	2.81e+02
1	Nitrite	3.48e+04	3.90e+04	4.40e+07	7	-1.99e+04	1.15e+04
l i	Nitrate	3.40e+04	3.70e+04	9.11e+07	l (-2.75e+04	2.16e+04
!	Phosphate	2.05e+04	2.21e+04	1.16e+08	5 7	-2.73e+04 -2.71e+04	2.10e+04 2.39e+04
i '	Suifate	6.50e+03	7.30e+03	3.80e+06	5	-5.81e+03	4.21e+03
l i	Total CN	4.41e+03	5.51e+03	2.89e+07	3	-2.42e+04	2.20e+04
i l	Sr-90	1.75e+03	1.18e+03	1.09e+06	ا ا	-1.79e+03	2.93e+03
\	Cs-137.fus	6.85e+02	8.74e+02	1.19e+05	2 9 5	-1.07e+03	6.98e+02
} -	00 137 . 700	1 0.076.05	3.746.02	1.176.03		-1.016403	0.700702
48	ICP.fus.Al	8.56e+03	8.57e+03	6.53e+09	4	-2.24e+05	2.24e+05
1 - 3	ICP.fus.Ca	2.30e+04	1.77e+04	5.84e+07	ו לי ו	-1.27e+04	2.35e+04
	ICP.fus.fe	2.05e+04	2.22e+04	2.12e+08	8	-3.52e+04	3.18e+04
1	ICP.fus.Na	1.09e+05	1.00e+05	5.42e+08	7	-4.62e+04	6.39e+04
1 1	ICP. fus.Pb	6.93e+02	NA NA	4.83e+07	NA	NA	NA NA
	ICP.fus.U	1.55e+04	2.47e+04	1.18e+08	2	-5.60e+04	3.76e+04
1	ICP.fus.P	2.21e+04	2.02e+04	4.63e+07	9	-1.35e+04	1.73e+04
1 1	Chloride	9.75e+02	8.00e+02	2.08e+04	5	-1.96e+02	5.46e+02
i l	Nitrite	5.03e+04	4.50e+04	5.95e+07	6	-1.36e+04	2.41e+04
	Nitrate	5.48e+04	4,80e+04	1.15e+08	5	-2.08e+04	3.43e+04
]]	Phosphate	2.59e+04	2.67e+04	1.49e+08	8	-2.90e+04	2.73e+04
l i	Sulfate	1.05e+04	9.25e+03	4.73e+06	5	-4.34e+03	6.84e+03
i I	Total CN	9.95e+03	1.44e+04	3.05e+07	3	-2.20e+04	1.32e+04
]]	\$r-90	1.36e+02	1.90e+02	1.50e+06	9	-2.82e+03	2.72e+03
j i	Cs-137. fus	1.16e+03	1.03e+03	1.49e+05	5	-8.64e+02	1.12e+03
							10.120.00
49	ICP.fus.Al	1.17e+05	1.27e+05	5.79e+09	3	-2.51e+05	2.33e+05
1	ICP.fus.Ca	1.51e+04	1.49e+04	4.75e+07	6	-1.66e+04	1.71e+04
	ICP.fus.Fe	1.19e+04	9.10e+03	1.60 e+ 08	8	-2.64e+04	3.19e+04
1 1	ICP.fus.Na	6.58e+04	7.65e+04	4.08e+08	7	-5.85e+04	3.70e+04
]	ICP, fus.Pb	1.36e+03	8.23e+02	3.45e+07	3	-1.82e+04	1.92e+04
1 1	ICP.fus.U	1.24e+04	5.59e+03	1.18e+08	2	-4.00e+04	5.36e+04
[]	ICP.fus.P	1.21e+04	1.46e+04	3.41e+07	19	-1.57e+04	1.08e+04
	Chloride	7.00e+02	7.00 e+ 02	1.50e+04	5 7	-3.14 e+ 02	3.14e+02
	Nitrite	3.83e+04	3.85e+04	4.40e+07	7	-1.59e+04	1.55e+04
1]	Nitrate	3.69e+04	3.60e+04	9.11e+07	5 7	-2.36e+04	2.54e+04
]	Phosphate	1.34e+04	1.28e+04	1.16e+08	7	-2.49 e+ 04	2.61e+04
[[Sulfate	6.97e+03	6.55e+03	3.80e+06	5	-4.59e+03	5.43e+03
	Total CN	5.71e+03	5.62e+03	2.89e+07	2	-2.30e+04	2.32e+04
]	\$r-90	9.28e+02	9.32e+02	1.09e+06	9	-2.36e+03	2.36e+03
	Cs-137.fus	3.89e+02	5.57e+02	1.19e+05	5	-1.05e+03	7.19e+02

Table 8. 95% Confidence Interval on $\sigma^2(A)$, Analytical Error Variance.

Analyte	ô ² (A)	df	95% LL	95% UL
ICP.acid.Al	1.00e+08	7	3,21e+07	1.39e+09
ICP.acid.Ca	3.91e+06	1 3	1.25e+06	5.43e+07
ICP.acid.Fe	5.45e+07	1 4	1.75e+07	7.57e+08
ICP.acid.Na	1.20e+08	1 1	5.89e+07	2.55e+09
ICP.acid.Ni	1.47e+06	1 1	4.72e+05	2.04e+07
ICP.acid.Pb	1.23e+06	1 1	3.96e+05	1.71e+07
ICP.acid.U	4.18e+06	1 3	1.34e+06	5.81e+07
ICP.acid.P	2.75e+07	3 3 3 3 3 3	1.41e+07	6.09e+08
131 (43 141)			10.7.0	0.070.00
ICP.fus.Ai	3.71e+07	3	1.19e+07	5.15e+08
ICP.fus.Ca	6.47e+05	3	2.08e+05	8.98e+06
ICP.fus.Fe	3.38e+06	3	1.08e+06	4.70e+07
ICP.fus.Na	4.97e+07	3	1.59e+07	6.90e+08
ICP.fus.Pb	3.71e+03	2	1.00e+03	1.47e+05
ICP.fus.U	9.45e+06	3 3 3 2 2	2.57e+06	3.74e+08
ICP.fus.P	9.43e+06	3	3.03e+06	1.31e+08
ICP.water.Ca	1.67e+02	3	5.35e+01	2.32 e+ 03
ICP.water.Fe	6.20e+02	3	1.99e+02	8.62e+03
ICP.water.Na	2.40e+07	3	7.70e+06	3.33e+08
ICP.water.Ni	1.66e+02	3 3 3 3	5.33e+01	2.31e+03
ICP.water.P	7.27e+06	3	2.33e+06	1.01e+08
Chloride	0.00	٠ - ٦	0.00	0.00
Nitrite	6.83e+06	-	2.19e+06	9.49e+07
Nitrate	6.67e+06	1	2.14+06	9.26e+07
Phosphate	5.93e+07	ं र	1.90e+07	8.24e+08
Sulphate	1.70e+05	3	5.45e+04	2.36e+06
Total CN	2.14e+04	3 3 3 3	6.88e+03	2.98e+05
			01000-03	21704.03
U (#g/g)	4.05e+00	3	1.30e+00	5.63e+01
Tot.Alpha.Pu	3.57e-03	3	1.15e-03	4.96e-02
Sr-90	1.24e+04	3	3.98e+03	1.72e+05
Pu-239/240	3.57e-03	3	1.15e-03	4,96e-02
Cs-137.water	1.35e+00	3 3 3 3 3	4.34e-01	1,88e+01
Cs-137.fusion	4.23e+03	3	1.36e+03	5.87e+04

Table 9. 95% Confidence Interval on $\sigma^2(S)$, Spatial Variance.

Analyte	∂ ² (\$)	df	p-value	95% LL	95% UL
ICP.acid.Al	1.70e+09	2	0.008	2.57e+08	6.91e+10
ICP.acid.Ca	1.72e+07	2 1	0.048	0.00	7.54e+08
ICP.acid.Fe	9.14e+07	2	0.130	0.00	4.66e+09
ICP.acid.Na	0.00	2 2 2 2 2	0.882	0.00	3.84e+08
ICP.acid.Ni	2.95e+06	2	0.110	0.00	1.45e+08
ICP.acid.Pb	2.00e+07	2 2 2 2	0.009	2.92e+06	8.16e+08
ICP.acid.U	1.71e+07	2	0.053	0.00	7.57e+08
ICP.acid.P	0.00	2	0.939	0.00	3.40e+07
ICP.fus.Al	4.27e+09	2	0.001	1.08e+09	1.69e+11
ICP.fus.Ca	2.52e+07	2 1	0.003	5.50e+06	1.01e+09
ICP.fus.Fe	5.39e+07	2	0.009	7.73e+06	2.19+09
ICP.fus.Na	1.14e+08	l ž l	0.097	0.00	5.48e+09
ICP.fus.Pb	2.08e+07	1 1	0.000	4,13e+06	2.13e+10
ICP.fus.U	9.80e+07	lżl	0.052	0.00	4.26e+09
ICP.fus.P	5.16e+06	2 2	0.270	0.00	3.85e+08
ICP.water.Ca	4.40e+03	2	0.004	8.52e+02	1.77e+05
ICP.water.fe	1.95e+04	l ž	0.003	4.03e+03	7.83e+05
ICP.water.Na	1.32e+08	2	0.037	0.00	5.70e+09
ICP.water.Ni	2.31e+03	1 5 1	0.011	2.89e+02	9.45e+04
ICP.water.P	1.60e+06	2 2	0.364	0.00	2.03e+08
Chloride	3.33e+03	2	0.000	9.03e+02	1.32e+05
Nitrite	9.67e+06	2	0.149	0.00	5.13e+08
Nitrate	4.10e+07	2	0.032	0.00	1.75e+09
Phosphete	2.08e+07	2	0.321	0.00	1.96e+09
Sulphate	1.86e+06	2	0.015	1.58e+05	7.66e+07
Total CN	2.52e+07	1 2	0.000	6.78e+06	9.94e+08
		1		0.700-00	7.746700
U (μg/g)	1.09e+02	2	0.004	2.12e+01	4.37e+03
Tot.Alpha.Pu	2.13e-01	2	0.001	5.06e-02	8.50e+00
sr-90 °	2.57e+05	2	0.006	4.45e+04	1.04e+07
Pu-239/240	2.13e-01	2	0.001	5.04e-02	8.48e+00
Cs-137.water	4.66e+00	2	0.064	0.00	2.10e+02
Cs-137, fusion	5.63e+04	121	0.012	6.66e+03	2.30e+06

Table 10. Tukey's HSD Multiple Comparisons, Core Composite Data (Units $\mu g/g$ Except Radionuclides $\mu Ci/g$).

		Group			Mean			
Core	47	48	49	47	48	49	RSD	
ICP.acid.Al	ß	Œ	ē	7.3e+04	6.4e+03	8.4e+04	18%	
ICP.acid.Ca	α	Œ	α	2.0e+04	1.3e+04	1.2e+04	13%	
ICP.acid.Fe	α	α	α	2.9e+04	2.0e+04	7.1e+03	39%	
ICP.acid.Na	α	α	α	8.2e+04	8.8e+04	8.1e+04	16%	
ICP.acid.Ni	· ·	α	α	1.5e+04	1.6e+04	1.2e+04	9%	
ICP.acid.Pb	α	₿	β	8.6e+03	6.1e+02	8.6e+02	33%	
ICP.acid.U	αβ	β	α	1.1e+04	1.5e+04	6.3e+03	19%	
ICP.acid.P	α	α	α	1.8e+04	1.7e+04	1.9e+04	36%	
ICP.fus.Al		a		1.2e+05	8.6e+03	1.3e+05	7%	
ICP.fus.Ca	l P	a a	βα	2.5e+04	1.8e+04	1.5e+04	42	
ICP.fus.Ca	2	Ä	a	2.2e+04	2.2e+04	9.1e+03	10%	
ICP.fus.Na	ا م	a	a	8.7e+04	1.0e+05	7.7e+04	8%	
ICP.fus.Pb	ا ت	-	α	7.3e+03	NA NA	8.2e+02	12	
ICP.fus.U	١ ۵	α	ā	9.2e+03	2.5e+04	2.8e+03	28%	
ICP.fus.P	a	ά	a l	2.0e+04	2.0e+04	1.5e+04	17%	
ICF. IUS.F				2.00,04	2.00.04	1.30.04	 ''~	
1CP.water.Ca	8	α	α	1.8e+02	6.0e+01	7.8e+01	12%	
ICP water Fe	ďα	В	α	8.8e+02	1.1e+03	9.2e+02	3%	
ICP.water.Na	αβ	8	α	6.8e+04	8.4e+04	6.0e+04	7%	
ICP water Ni	ß	à	α	1.2e+02	3.1e+01	5.3e+01	19%	
ICP.water.P	ά	α	α	7.0e+03	8.7e+03	4.2e+03	41%	
-4.4. 1.1				7.002	0.000	7.000	2	
Chloride	α	B	α	7.0e+02 3.9e+04	8.0e+02	7.0e+02 3.9e+04	0%	
Nitrite	α	a	α		4.5e+04		6%	
Nitrate	} a	P	, a	3.7e+04	4.8e+04	3.6e+04	6%	
Phosphate	α	α .	α	2.2e+04	2.7e+04	1.3e+04	38%	
Sulphate	a	1 P	a	7.3e+03 2.8e+03	9.3e+03 2.1e+02	6.6e+03 2.8e+03	5% 167%	
Total CN	<u> </u>	<u> </u>	<u> </u>	∠.oe+u3	Z. 1870Z	∠.8e+U3	10/4	
U(μg/g)	۱	B	α	1.2e+01	2.8e+01	7.5 e+ 00	13%	
Tot.Alpha.Pu	l ē	ا ا	α	8.8e-01	6.8e-02	7.9e-02	18%	
Sr-90	8	ا مَ	Ä	1.2e+03	1.9e+02	9.3e+02	15%	
Cs-137.water	ا و	α	ά	9.2e+00	9.3e+00	5.3e+00	15%	
Cs-137.fusion	l ä	l ē	ā	8.7e+02	1.0e+03	5.6e+02	8%	

Table 11. Tukey's HSD Multiple Comparisons, Subsegment Data (Units $\mu g/g$ Except Radionuclides $\mu Ci/g$).

		Group	·		Hean		
Core	47	48	49	47	48	49	RSD
ICP.fus.Al	€ € β	α α	\$ \$ Y	1.3e+05 1.2e+05 3.2e+04	7.3e+03 9.8e+03	1.9e+05 9.6e+04 7.1e+04	6 x
ICP.fus.Ca	β Υ «	e Y	α γδ δ	1.0e+04 1.8e+04 2.8e+04	2.9e+04 1.7e+04	4.3e+03 1.9e+04 2.3e+04	6%
1CP.fus.Fe	β α α	a a	a a a	6.3e+04 2.1e+04 1.5e+04	2.0e+04 2.1e+04	1.6e+04 4.6e+03 1.5e+04	46X
ICP.fus.Na	α β αβγ γδ	δ γδ	α αβγ βγδ	5.1e+04 6.3e+04 1.0e+05	1.2e+05 1.0e+05	4.3e+04 6.3e+04 9.1e+04	14%
ICP.fus.Pb	α α α	- a	α - α	5.0e+03 2.9e+03 1.4e+04	NA 6.9 e+ 02	2.0e+03 NA 7.3e+02	50%
ICP.fus.U	γδ αβ α	ε δε	αβγ γδ	1.2e+04 6.1e+03 5.8e+03	1.7e+04 1.4e+04	7.9e+03 NA 1.2e+04	9%
ICP.fus.P	- αβ δ	Y	- α βγ	NA 1.2e+04 3.0e+04	2.3e+04 2.1e+04	NA 1.1e+04 2.0e+04	9%
Chloride	αβ βγ Υ	δε ε	α γδ γδ	5.5e+02 7.0e+02 7.5e+02	9.5e+02 1.0e+03	5.0e+02 8.0e+02 8.0e+02	6%
Nitrite	α β βγ	€ δ€	α γ γδ	2.8e+04 3.7e+04 4.0e+04	5.1e+04 5.0e+04	2.6e+04 4.4e+04 4.5e+04	4%
Nitrate	а в ув	δ δ	α βγ Υ	2.8e+04 3.6e+04 3.9e+04	5.6e+04 5.4e+04	2.6e+04 4.2e+04 4.3e+04	4%
Phosphate	a a Y	а в Ву	α α αβγ	7.3e+03 9.6e+03 4.5e+04	1.6e+04 3.6e+04	6.1e+03 8.8e+03 2.5e+04	28%
Sulfate	α β βγ	€ 5	œ Y Y	5.1e+03 7.1e+03 7.4e+03	1.1e+04 1.0e+04	4.7e+03 8.2e+03 8.1e+03	3%
Total CN	æ ß Y	€ 8	æ S Y	3.2e+03 4.4e+03 5.8e+03	1.1e+04 8.7e+03	3.5e+03 8.1e+03 5.5e+03	3%
Sr-90	δ φ αβ	α β α	Υ αβ αβ	4.6e+03 4.7e+02 2.2e+02	1.5e+02 1.2e+02	2.4 e+ 03 2.0 e+ 02 1.9e+02	8%
Cs-137.fusion	αβ βγδ γδ	& &	α αβ βγ	3.4e+02 7.7e+02 9.5e+02	1.2e+03 1.2e+03	1.2e+02 3.5e+02 7.0e+02	16%

Table 12. Comparison of Analytical Error Variances, 241-C-109 and 241-C-112.

Tank	C-109		C-112		
Analyte	δ ² (A)	df	δ ² (A)	df	p-value
ICP.acid.Al	1.00e+08	3	3.10e+07	3	0.180
ICP.acid.Ca	3.91e+06	3	1.44e+06	3	0.217
ICP.acid.Fe	5.45e+07	3	2.76e+07	3	0.294
ICP.acid.Na	1.20e+08	3 3 3 3 3 3 3	2.39e+07	3 3 3 3 3 3	0.294
ICP.acid.Ni	1.47e+06	3	1.06e+06	[3	0.399
ICP.acid.Pb	1.23e+06	3	2.41e+04	3	0.004
ICP.acid.U	4.18e+06	3	1.10e+08	3	0.012
ICP.acid.P	2.75e+07	3	3.83e+06	3	0.070
ICP.fus.Al	3.71e+07	3	5.67e+05	2	0.015
ICP.fus.Ca	6.47e+05	3	1.56e+05	1 2	0.200
ICP.fus.Fe	3.38e+06	3 3 3 2 2 2	9.81e+06	1 5	0.192
ICP.fus.Na	4.97e+07	3	3.96e+06	1 2	0.075
ICP.fus.Pb	3.71e+03	<u> </u>	1.47e+05	[5	0.025
ICP.fus.U	9.45e+06	l ž	1.64e+07	1 5	0.366
ICP.fus.P	9.43e+06	3	1.37e+05	2 2 2 2 2 2 2 2	0.014
ICP.water.Al	l NA	NA.	3.05e+04	,	NA
ICP.water.Ca	1.67e+02		3.78e+04	1 2	0.000
ICP.water.Fe	6.20e+02	3 3 3	3.43e+03	1 2	0.000
ICP.water.Na	2.40e+07	J 3	5.11e+08	5	0.017
ICP.water.Ni	1.66e+02	1 3	1.29e+03	5	0.065
ICP.water.U	Í NA	NA	2.60e+06	1 5	NA
ICP.water.P	7.27e+06	3	1.13e+07	2 2 2 2 2 2	0.345
Chloride	NA NA	NA	2.50e+04	2	NA
Nitrite	6.83e+06		1.33e+08	2 2 2 2 2	0.019
Nitrate	6.67e+06	3 3 3 3	2.41e+08	1 5	0.008
Phosphate	5.93e+07	3	1.10e+08	5	0.298
Sulphate	1.70e+05	3	6.25e+06	5	0.008
Total CN	2.14e+04	3	NA	NA.	NA NA
J (#g/g)	4.05e+00	3	5.30e+07	2	0.000
Tot.Alpha.Pu	3.57e-03	1 3	NA NA	l ĥa l	NA
Sr-90	1.24e+04	3 3 3	1.36e+03	2	0.101
Pu-238	NA.	NA NA	1.60e-05	2	NA NA
Pu-239/240	3.57e-03		2.69e-05	2	0.007
Cs-137.water	1.35e+00	3	NA NA	I ÑA I	NA
Cs-137.fusion	4.23e+03	3	2.31e+02	2 1	0.001

Table 13. Comparison of Spatial Variances, 241-C-109 and 241-C-112.

Tank	C-109		€-112		
Analyte	∂ ² (\$)	df	∂ ² (\$)	df	p-value
ICP.acid.Al	1.70e+09	2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	3.67e+08	2	0.178
ICP.acid.Ca	1.72e+07] 2	2.49e+07	2	0.408
ICP.acid.fe	9.14e+07	2	0.00	2 2 2 2	NA NA
ICP.acid.Na	0.00	1 2	2.66e+08	<u>2</u>	NA.
ICP.acid.Ni	2.95e+06	2	1.55e+07	2	0.160
ICP.acid.Pb	2.00e+07	4	2.28e+06	2 2 2	0.102
ICP.acid.U	1.71e+07 0.00	1 5	1.56e+09 4.52e+07	{	0.011
ICP.acid.P	0.00		4.5Ze+U/	۷	NA NA
ICP.fus.Al	4.27e+09	,	4.70e+08	,	0.099
ICP.fus.Ca	2.52e+07	2 2 2 2	4.09e+07	2 2 2 2 2 2 2	0.382
ICP.fus.Fe	5.39e+07	5	1.65e+07	5	0.234
ICP.fus.Na	1.14e+08	2	5.24e+08	2	0.179
ICP.fus.Pb	2.08e+07	l ī	3.98e+06	2	0.149
ICP.fus.U	9.80e+07	ĺż	1.78e+09	. Ž	0.052
ICP.fus.P	5.16e+06	2 2	8.04e+07	2	0.060
101110011				-	7.707
ICP.water.Al	l NA	NA NA	7.06e+04	2	NA NA
ICP.water.Ca	4.40e+03	2	0.00	2	NA NA
ICP.water.Fe	1.95e+04	2 2 2 2	2.26e+05	2 2 2 2 2 2 2	0.079
ICP.water.Na	1.32e+08	2	5.01e+08	2	0.209
ICP.water.Ni	2.31e+03		8.56e+04	2	0.026
ICP.water.U	NA NA	NA NA	4.55e+06	2	NA
ICP.water.P	1.60e+06	2	3.50e+07	2	0.044
Chioride	3.33e+03	,	5.25e+04	2	0.060
Nitrite	9.67e+06	5	1.04e+08	2	0.085
Nitrate	4.10e+07	5	2.21e+08	2	0.156
Phosphate	2.08e+07	5	2.97e+08	2	0.065
Sulphate	1.86e+06	5	9.17e+06	2	0.168
Total CN	2.52e+07	2 2 2 2 2 2	NA NA	NA	NA NA
		 	 		
U (μg/g)	1.09e+02	2	2.88e+09	1	0.000
Tot.Alpha.Pu	2.13e-01	2 2 2	NA	NA	NA
sr-90	2.57e+05	2	2.96e+06	2	0.080
Pu-238	NA NA	ŇA	7.81e-03	Ž	NA
Pu-239/240	2.13e-01	2	4.15e-03	2	0.019
Cs-137.water	4.66e+00	2 2 2	NA	NA	NA NA
Cs-137.fusion	5.63e+04	2	2.47e+03	2	0.042

Table 14. Comparison of Percent Variance, 241-C-109 and 241-C-112.

Tank	C-109	C-112	C-109	C-112
Analyte	%∂ ² (A)	%∂ ² (Α)	%ð ² (\$)	%∂ ² (\$)
ICP.acid.AL	6%	8%	94%	92%
ICP.acid.Ca	19%	5 %	81%	95%
ICP.acid.Fe	37%	100%	63%	0%
ICP.acid.Na	100%	8%	0%	92%
ICP.acid.Ni	33%	6%	67%	94%
ICP.acid.Pb	6%	12	94%	99%
ICP.acid.U	20%	7%	80%	93%
1CP.acid.P	100%	8%	0%	92%
ICP.fus.At	1%	0%	99%	100%
ICP.fus.Ca	32	0%	97%	100%
ICP.fus.Fe	6%	37%	94%	63%
ICP.fus.Na	30%	1 12	70%	99%
ICP.fus.Pb	0%	4%	100%	96%
ICP.fus.U	9%	1%	91%	99%
ICP.fus.P	65%	0%	35%	100%
ICP.water.Al	NA NA	30%	, NA	70%
ICP.water.Ca	4%	100%	96%	0%
ICP.water.Fe	3%	1%	97%	99%
ICP.water.Na	15%	51%	85%	49%
ICP.water.Ni ICP.water.U) 7% NA	1%	93%	99%
ICP.water.D	82%	36% 24%	NA 1997	64%
TCF.Water.F	02.5	24%	18%	76%
Chloride	0%	32%	100%	68%
Nitrite	41%	56%	59%	44%
Nitrate	14%	52%	86%	48%
Phosphate	74%	27%	26%	73%
Sulphate	8%	41%	92%	59%
Total CN	0%	NA NA	100%	NA .
U (μg/g)	4%	2%	96%	98%
Tot.Alpha.Pu	2%	NA.	98%	NA
Sr-90	5%	0%	95%	100%
Pu-238	NA 200	0%	NA OOW	100%
Pu-239/240 Cs-137.water	2%	1%	98%	99%
	22%	NA Ov	78%	NA CAS
Cs-137.fusion	7%	9%	93%	91%

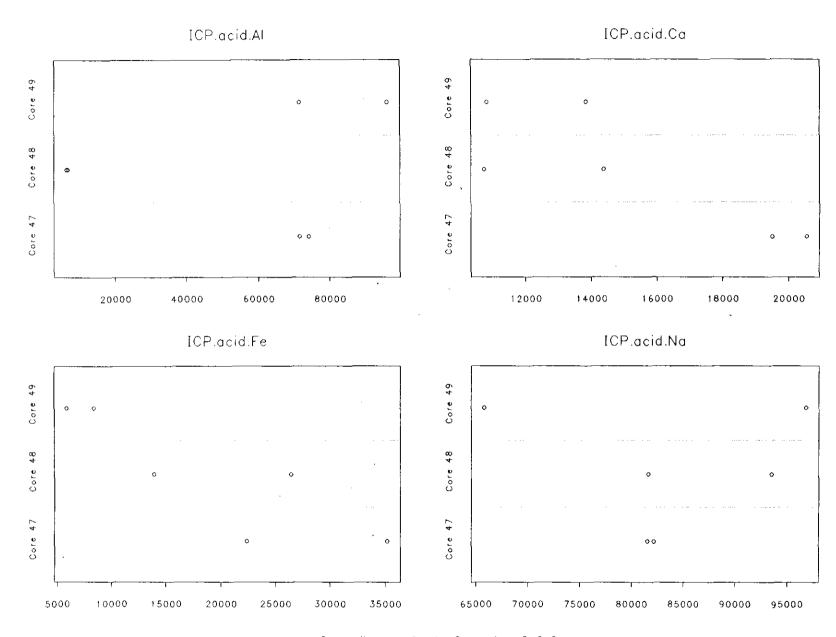
Table 15. Relative Standard Deviations from Tanks C112 and C109.

Tank	C-109	C-112	C-109	C-112	C-109	C-112
Analyte	RSD(ÿ)	RSD(ÿ)	RSD(A)	RSD(A)	RSD(S)	RSD(S)
ICP.acid.Al	44%	46%	18%	23%	76%	78%
ICP.acid.Ca	17%	17%	13%	7%	28%	30%
ICP.acid.Fe	34%	10%	39%	24%	51%	0%
ICP.acid.Na	2%	11%	13%	5%	0%	18%
ICP.acid.Ni	8%	18%	9%	8%	12%	30%
ICP.acid.Pb	78%	34%	33%	6%	133%	59%
ICP.acid.U	24%	40%	19%	18%	39%	68%
ICP.acid.P	4%	17%	29%	9%	0%	29%
ICP.fus.Al	45%	46%	7%	3%	78%	024
ICP.fus.Ca	15%	18%	42	2%	26%	82% 32%
ICP. fus. Fe	24%	10%	10%	111%	41%	15%
ICP.fus.Na	8%	12%	8%	2 X	12%	22%
ICP.fus.Pb	80%	39%	2%	13%	113%	68%
ICP.fus.U	42%	30%	21%	5x	67%	52%
ICP.fus.P	10%	18%	17%	1%	12%	32%
ICP.water.Al	NA NA	33%	NA	34 x	NA	51%
ICP.water.Ca	. 36%	11%	12%	58%	62%	
ICP.water.Fe	8%	23%	3%	5%	14%	0% 41%
ICP.water.Na	10%	18%	7%	26%	16%	25%
ICP.water.Ni	41%	24%	19%	5%	69%	43%
ICP.water.U	NA I	65%	NA	74%	NA.	98%
ICP.water.P	20%	36%	41%	33%	19%	59%
Chloride	5 %	15%	0%	16%		074
Nitrite	5%	16%	6%	24%	8% 8%	23%
Nitrate	10%	18%	6%	25%	16%	22%
Phosphate	20%	35%	38%	34%	22%	24% 56%
Sulphate	10%	17%	5%	21%	18%	
Total CN	34%	NA	2%	NA NA	59%	25% NA
U (μg/g)	39%	52%	13%	11%		
Tot.Alpha.Pu	78%	NA	18%	NA I	66% 135%	78%
Sr-90	39%	44%	15%	2%		NA
Pu-238	NA I	110%	NA NA	6%	66%	79%
Pu-239/240	78%	86%	18%	9%	NA 135%	137%
Cs-137.water	17%	NA NA	15%	NA NA	27%	107%
Cs-137.fusion	17%	5%	8%	2%	29%	NA 7%

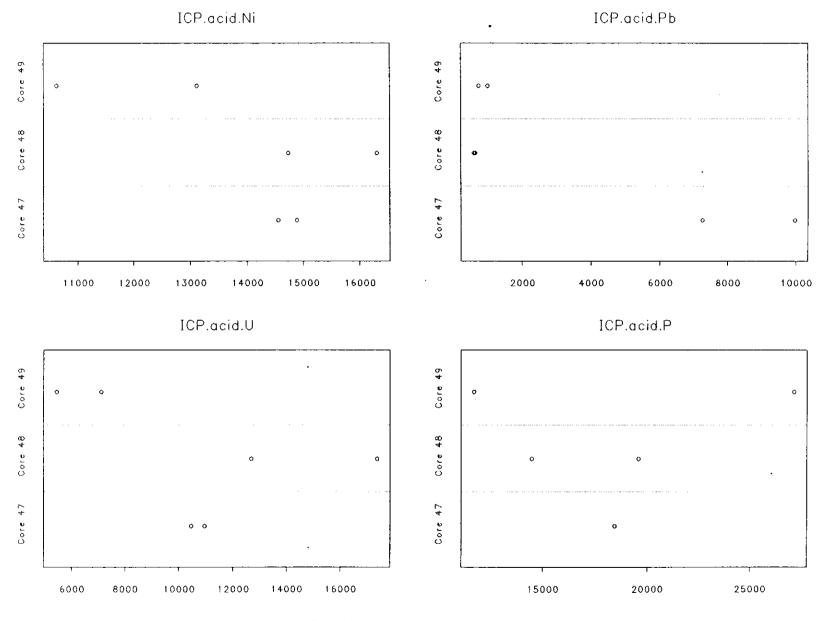
APPENDIX 2: C109 CORE COMPOSITE AND SUBSEGMENT DATA PLOTS

This appendix contains plots of the core composite and subsegment data for Clo9. The units for the analytes in the plots are as follows:

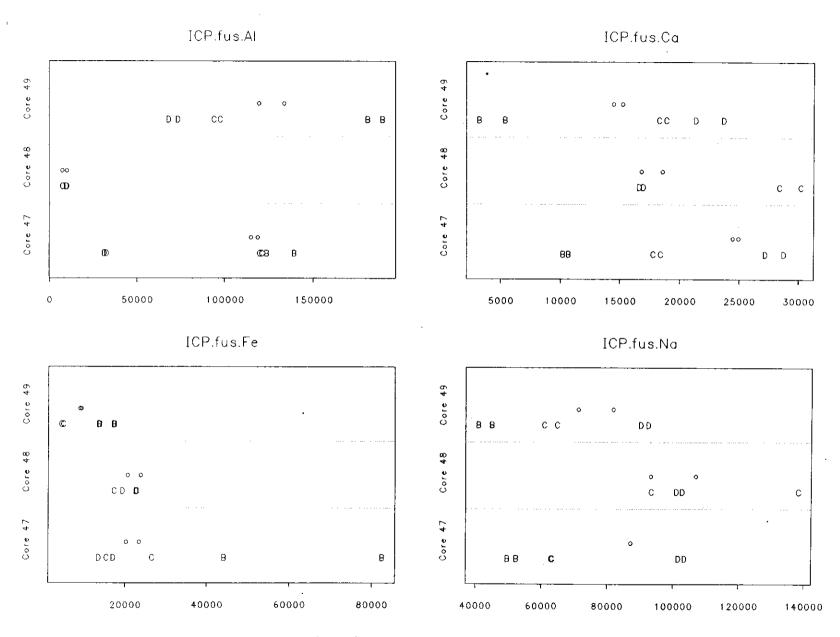
Analyte	Units	
All ICP (acid, water, fusion)	μg/g	
All Anions	μg/g	
All Radionuclides (excluding U)	μCi/g	
U	μg/g	



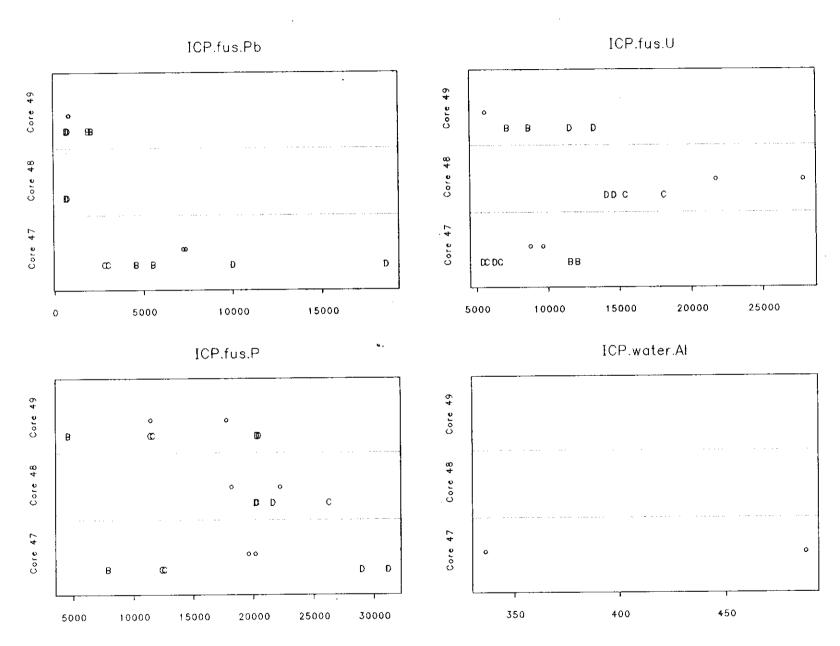
Composite = o, Quarter Segments = B, C, D



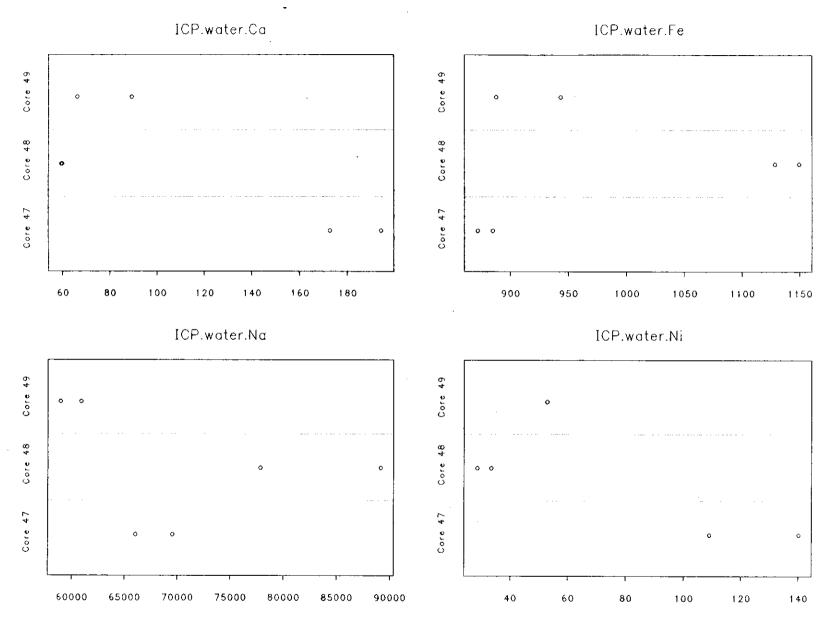
Composite = o, Quarter Segments = B, C, D



Composite = o, Quarter Segments = B, C, D

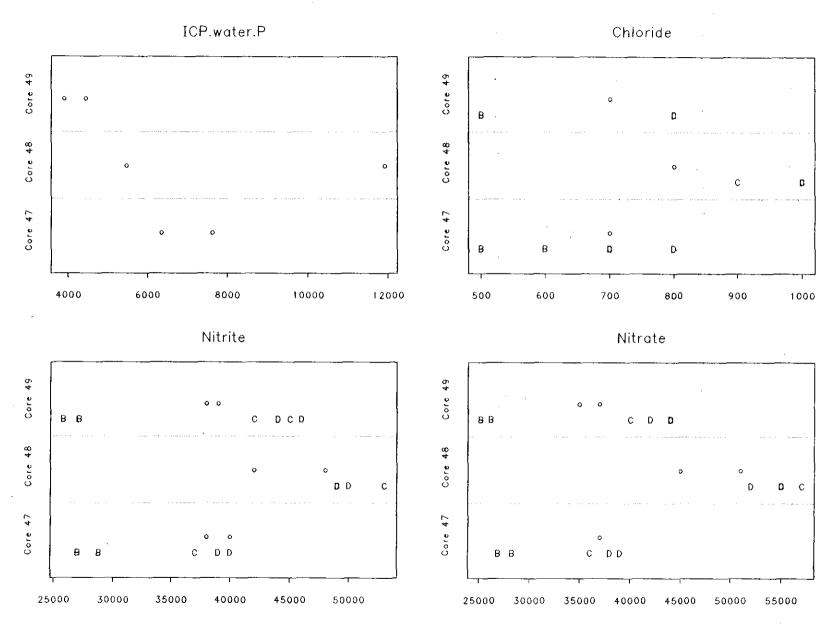


Composite = o, Quarter Segments = B, C, D

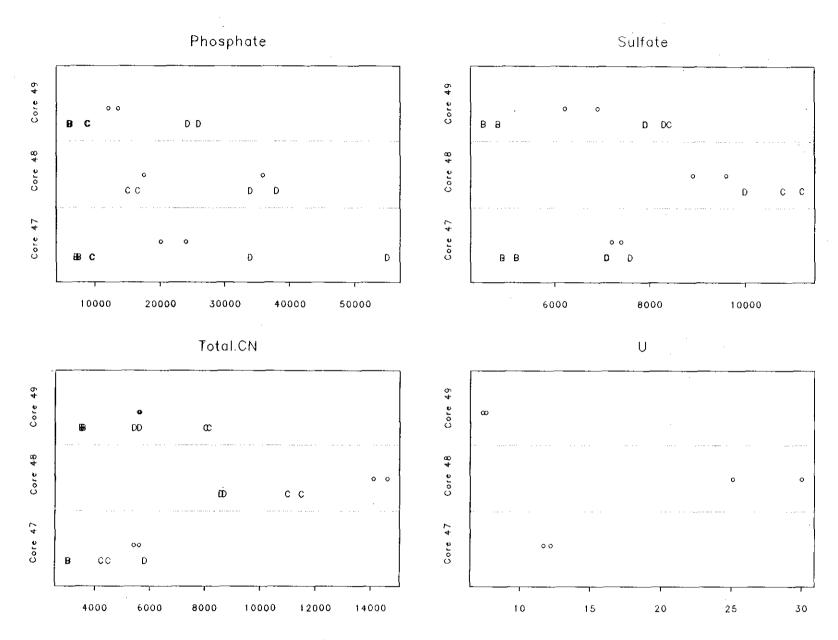


Composite = o_i Quarter Segments = B_i C_i D

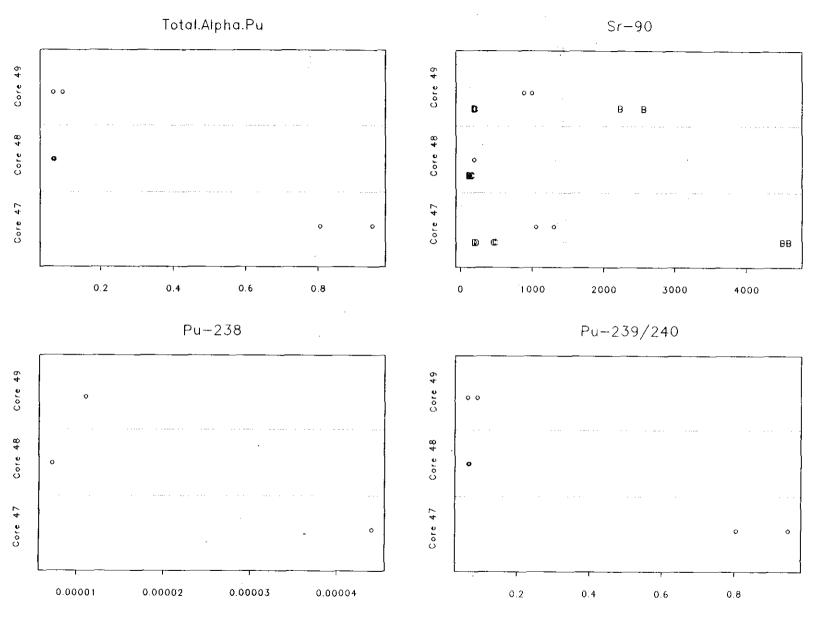
WHC-EP-0668



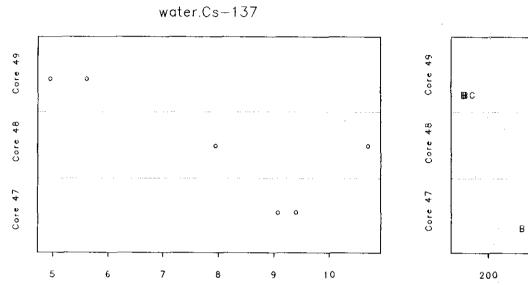
Composite = o, Quarter Segments = B, C, D

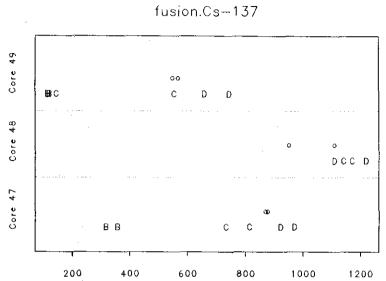


Composite = o, Quarter Segments = B, C, D



Composite = o, Quarter Segments = B, C, D



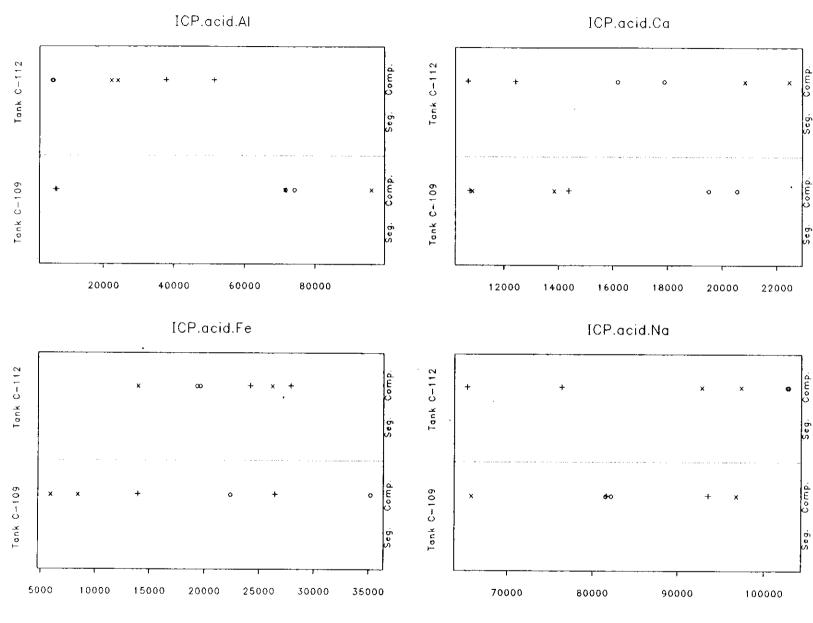


Composite = o, Quarter Segments = B, C, D

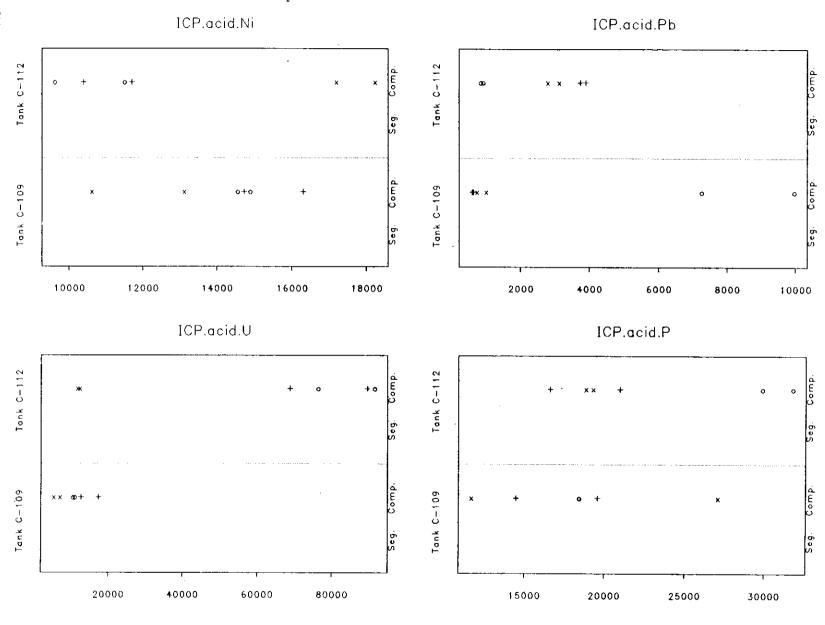
APPENDIX 3: C109 AND C112 CORE COMPOSITE AND SUBSEGMENT DATA PLOTS

This appendix contains plots of the core composite and subsegment data for Cl09 and Cl12. The units for the analytes in the plots are as follows:

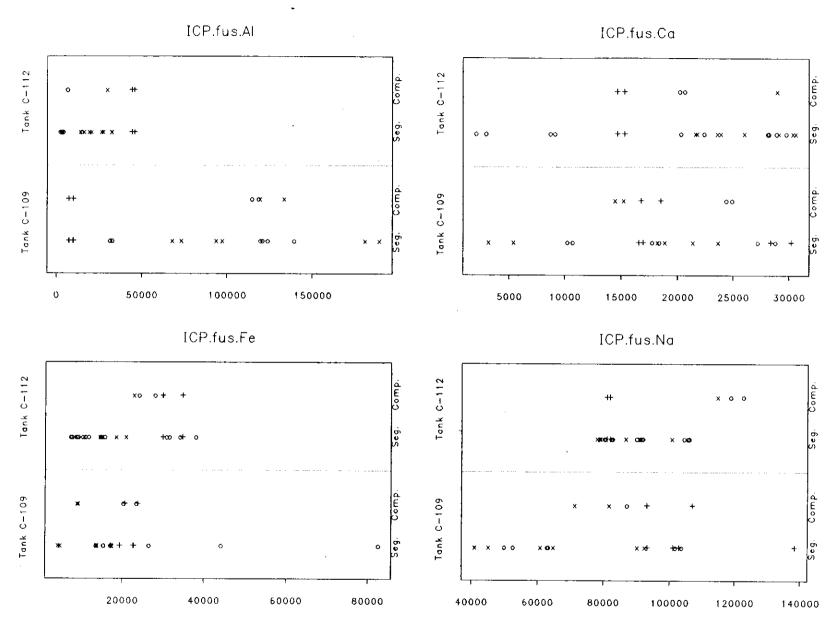
Analyte	Units
All ICP (acid, water, fusion)	μg/g
All Anions	μg/g
All Radionuclides (excluding U)	μCi/g
U	μg/g



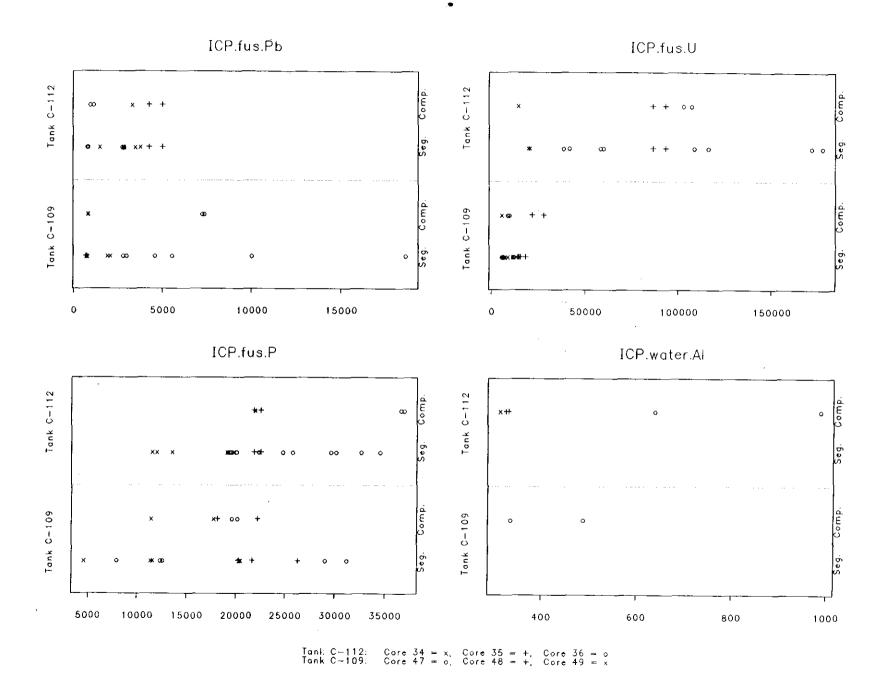
Tank C-112: Core 34 = x, Core 35 = +, Core 36 = 0 Tank C-109: Core 47 = 0, Core 48 = +, Core 49 = x

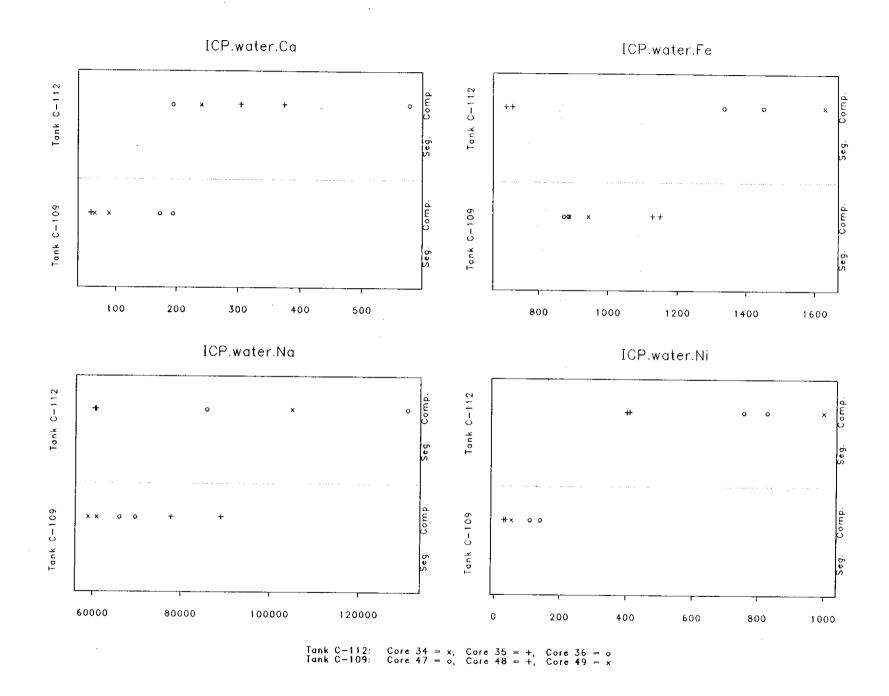


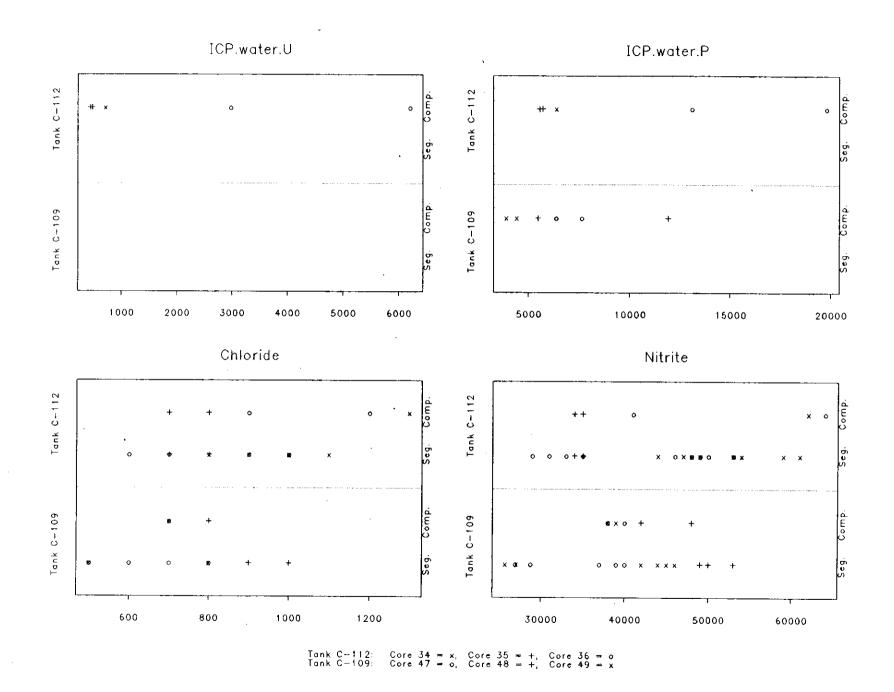
Tank C-112: Core 34 = x, Core 35 = +, Core 36 = 0Tank C-109: Core 47 = 0, Core 48 = +, Core 49 = x

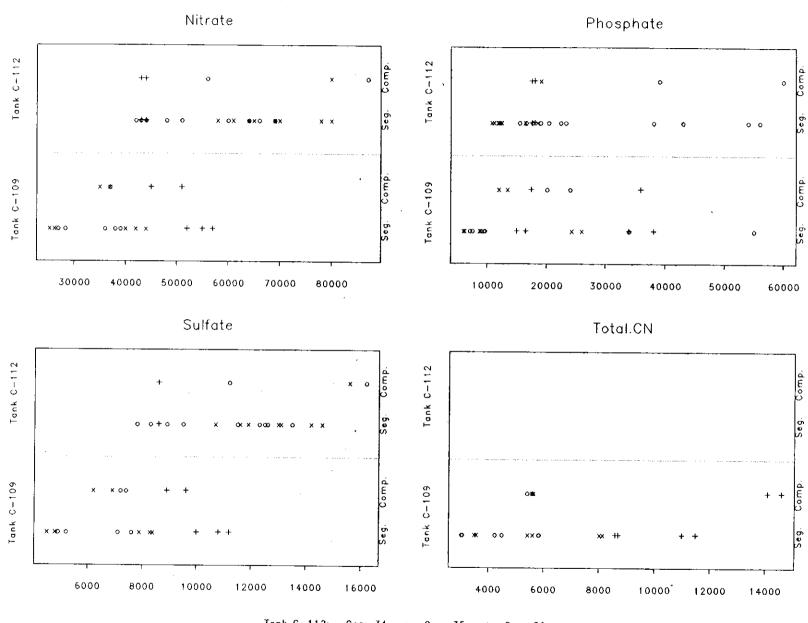


Tank C-112: Core 34 = x, Core 35 = +, Core 36 = 0Tank C-109: Core 47 = 0, Core 48 = +, Core 49 = x

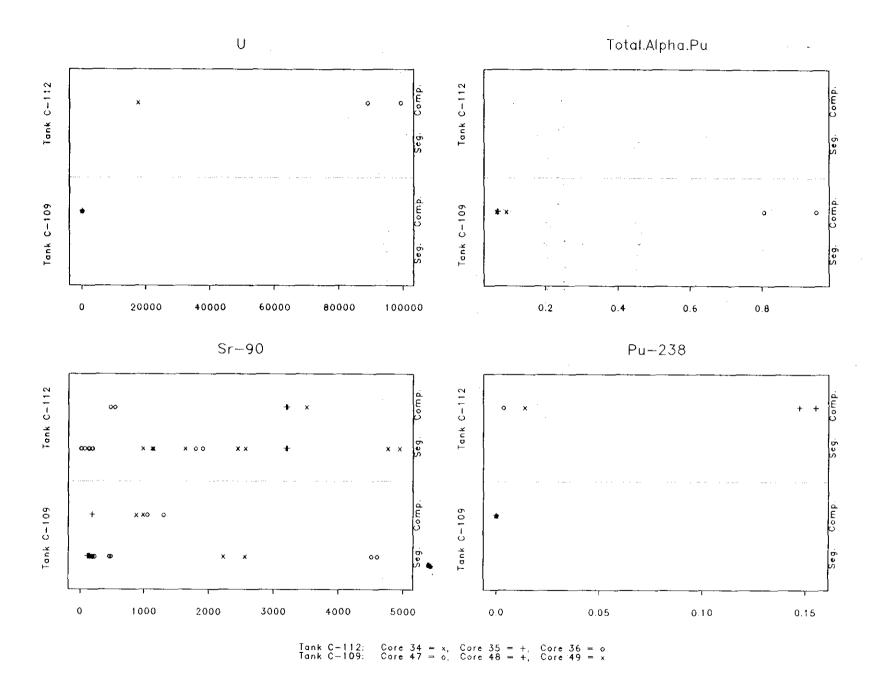


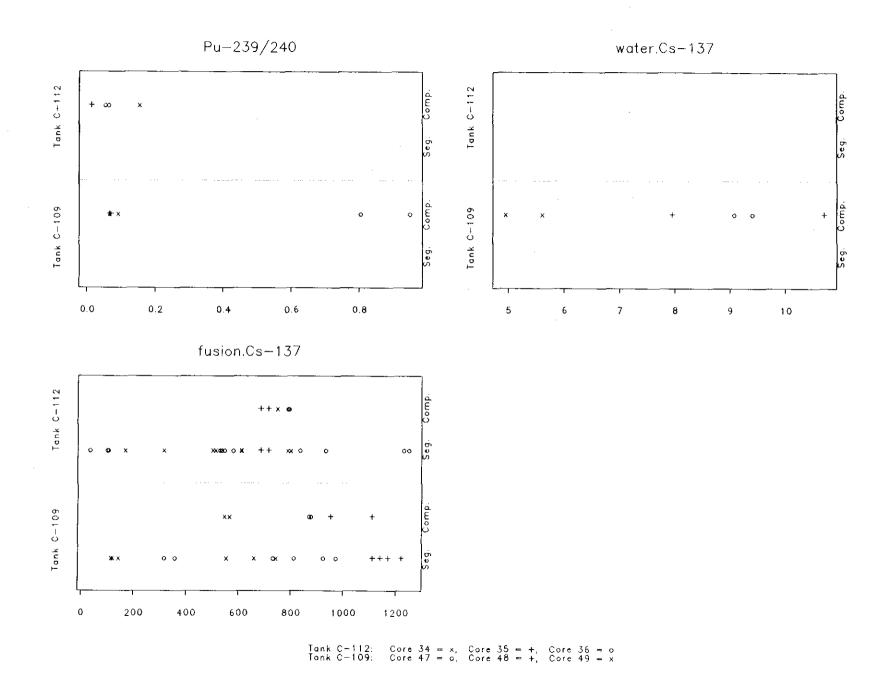






Tank C-112: Core 34 = x, Core 35 = +, Core 36 = 0Tank C-109: Core 47 = 0, Core 48 = +, Core 49 = x





APPENDIX 4: DERIVATIONS OF $\hat{\sigma}^2[\bar{y}(w)]$ AND $\hat{\sigma}^2[\bar{y}(w)-\bar{y}(c)]$

This appendix contains the derivations of $\hat{\sigma}^2[\bar{y}(w)]$ and $\hat{\sigma}^2[\bar{y}(w)-\bar{y}(c)]$. For the formulas in this appendix we define the following quantities: $\bar{y}(w)=\bar{y}_{wi}$ and $\bar{y}(c)=\bar{y}_{i}^{\dagger}$. That is, $\hat{\sigma}^2[\bar{y}(w)]=\hat{\sigma}^2[\bar{y}_{wi}]$ and $\hat{\sigma}^2[\bar{y}(w)-\bar{y}(c)]=\hat{\sigma}^2[\bar{y}_{wi}-\bar{y}_{i}^{\dagger}]$.

Three statistical models are used in the calculation of these two variance estimates. The first model, for the core and quarter segment data is

$$y_{ijk} = \mu + c_i + s_{ij} + e_{ijk}. {1}$$

The mean of the two aliquots from the j th quarter segment of the i th core is \bar{y}_{ij} . A statistical model for this mean is

$$\overline{y}_{ij} = \mu + c_i + E_{ij} \tag{2}$$

where

 $ar{y}_{ij.}$ = mean of the two aliquots from the jth quarter segment of the ith core,

 μ = overall mean of all the data,

c, = the effect of the ith core,

 $E_{ij} = s_{ij} + \bar{e}_{ij}$,

 s_{ij} = the effect of the jth quarter segment in the ith core, and

 \bar{e}_{ij} = the residual of the jth quarter segment mean in the ith core.

The subscripts i, j, and k have the following ranges: $i=1,\ldots,a$ (a=number of cores or core composites), $j=1,\ldots,b_i$ ($b_i=1,\ldots,b_i=1,\ldots$

The third model, for core composite sample data is

$$y'_{ik} = \mu' + c'_{i} + e'_{ik}$$
 (3)

where

 $y_{ik}^{t} = k^{th}$ aliquot of the i^{th} core composite,

 μ ' = the overall mean of the all core composite aliquots,

 $c_i^t = i^{th}$ core composite effect, and

 $e_{ik}^{t} = k^{th}$ residual of the i^{th} core composite.

The subscript ranges are the same as those used for the previous model.

The variance of \bar{y}_{ij} is

$$V(\overline{y}_{ij.}) = V(\mu + c_i + E_{ij})$$
$$= \frac{\sigma^2}{n} + \sigma_s^2 + \sigma_c^2$$

where σ_c^2 is the variance between cores, σ_s^2 is the variance between quarter segments and σ^2 is the analytical error.

A weighted mean representing the simulated core composite (SCC) is

$$\overline{y}_{wi} = \sum_{j=1}^{b_i} w_{ij} \overline{y}_{ij}.$$

The variance of $\boldsymbol{\bar{y}_{wi}}$ is

$$V(\overline{y}_{wi}) = V\left[\sum_{j=1}^{b_{i}} w_{ij} \ \overline{y}_{ij}\right] = \sum_{j=1}^{b_{i}} w_{ij}^{2} \ V(\overline{y}_{ij})$$
$$= \sum_{j=1}^{b_{i}} w_{ij}^{2} \left[\frac{\sigma^{2}}{n} + \sigma_{s}^{2} + \sigma_{c}^{2}\right].$$

The between mean squares (BMS) from model (2) has expectation

$$E(BMS) = \frac{\sigma^2}{n} + \sigma_s^2 + b_o \sigma_c^2$$

where

$$b_{o} = \frac{\sum_{i=1}^{a} b_{i}^{2}}{\sum_{i=1}^{a} b_{i}}.$$

The within mean squares (WMS) from model (2) has expectation

$$E(WMS) = \frac{\sigma^2}{n} + \sigma_s^2.$$

By solving a system of two equations with two unknowns, we obtain

$$\hat{\sigma}_{c}^{2} = \frac{BMS - WMS}{b_{o}},$$

$$\frac{\hat{\sigma}^{2}}{n} + \hat{\sigma}_{s}^{2} = WMS$$

as estimates of σ_c^2 and $(\sigma^2/n + \sigma_s^2)$ respectively.

From the results above we can obtain an estimate of $\sigma^2(\bar{y}_{wi})$, which is

$$\begin{split} \hat{\sigma}^{2}(\overline{y}_{wi}) &= \hat{V}(\overline{y}_{wi}) \\ &= \sum_{j=1}^{b_{i}} w_{ij}^{2} \left[\frac{\hat{\sigma}^{2}}{n} + \hat{\sigma}_{s}^{2} + \hat{\sigma}_{c}^{2} \right] \\ &= \sum_{j=1}^{b_{i}} w_{ij}^{2} \left[\frac{WMS + \frac{BMS - WMS}{b_{o}}}{b_{o}} \right] \\ &= \sum_{j=1}^{b_{i}} w_{ij}^{2} \left[\frac{BMS + (b_{o}-1)WMS}{b_{o}} \right]. \end{split}$$

The variance for \bar{y}_{i} , using the structure from model (3), is

$$V(\overline{y}_{i.}') = \frac{\sigma^{2'} + n\sigma_{c}^{2'}}{n}$$

where σ^2 is the analytical error and σ_c^2 is the variability between cores. The between mean squares (BMS') expectation for model (3) is

$$E(BMS') = \sigma^{2'} + n\sigma_c^{2'}.$$

Using this expectation, an estimate of the variance for $\mathbf{\bar{y}}_{i}^{\prime}$ is

$$\hat{V}(\overline{y}'_{i}) = \frac{BMS'}{n}$$
.

The estimated variance of $\boldsymbol{\bar{y}_{wi}} \boldsymbol{-} \boldsymbol{\bar{y}_{i}}$ is

$$\begin{split} \hat{\sigma}^2(\overline{y}_{\text{wi}} - \overline{y}_{\text{i.}}^{\,\prime}) &= \hat{\sigma}^2(\overline{y}_{\text{wi}}) + \hat{\sigma}^2(\overline{y}_{\text{i.}}) \\ &= \left[\sum_{j=1}^{b_i} w_{ij}^{\,2} \left[\frac{\text{BMS} + (b_o - 1) \text{WMS}}{b_o} \right] \right] + \left[\frac{\text{BMS}^{\,\prime}}{n} \right]. \end{split}$$

We have assumed that the covariance between the two means is zero.

This page intentionally left blank.

APPENDIX C

MISCELLANEOUS DATA

This page intentionally left blank.

MEETING MINUTES

	Subject: ENER	GY MEASUREMENTS	FOR DISQUA	LIFYING WA	STE TANKS	FROM WATCH	LISTS
ro: [Distribution	·	<u> </u>		SUILDING:	MO-037	····
ROH:	J. R. Jewett	ric .			· · · · · · · · · · · · · · · · · · ·		
eeting	Date .		Number Attend	iing			
10/22	1/92		6				
÷	H. Babad D. B. Bechtol M. L. Bell R. J. Cash D. A. Dodd D. J. Hert J. R. Jewett	R2-08* d T6-50* T6-16 R2-32 T6-50 T6-30* T6-50*	A. F. J. C. J. P. H. E. J. H.	Marshall Noonan Person Sloughter Smith Tillman Winters	R2-12* T6-30 T6-50	ХC	GTD DIU Jeppson DIU Jeppson DIU Jeppson RD CRING RD CRIPHEN RD CRIPHEN RD SCHEELE
	* Attendees		: 1992	3 C S C S C S C S C S C S C S C S C S C	_		GF Schiefers 10 Horrins 60 Horrins

Harry Babad is preparing to develop a criterion based on energetic measurements for removing tanks from the watch lists. He wants the labs to be prepared for the sample loads which may occur as a result of this criterion. Initial thoughts about the criterion are as follows:

If the exotherm of the material is < 75 cal/g, there should be no further concern. If moisture is > 25%, the limit of concern may be raised to 125 cal/g. These are figures determined by Babad in consultation with a number of nationally recognized experts in the field.

The suggestion is that if the differential scanning calorimetry exotherms exceed this level, then more in-depth examination, such as adiabatic calorimetry, would be necessary. Adiabatic calorimetry would give such information as initiation temperature, reaction rate, and propagation rate. Babad estimates that perhaps there might be 20 tanks that exceed these limits. (This number is, of course, open to debate.) There would also be need for measurements to support studies of synthetic materials.

A number of deficiencies of adiabatic calorimetry and thoughts about improvement were mentioned during the meeting. Keeping in spirit of the meeting, which was by and large a brain storming session, the ideas are presented here in no particular order.

Do we have enough tools? Adiabatic is slow, labor intensive, dose rate high.
 It uses too much sample; we have just one. Does the Accelerating Rate
 Calorimeter (ARC; by Columbia Scientific) offer any advantage over the Fauske
 Reactive System Screening Tool? If it is too big, could it be modified?

54-3000-100 (4/58) (EF) GEF011 Heating Minutes

- 2. Do we have the right tools to work up the results? We should have a bigger computer. Would Chemometric tools help? (Eric Wyse at PNL may have an idea.) Could data work-up be more automated?
- 3. Do we have enough capacity?
- 4. On we have proper documentation for our measurements? We use test plans and test reports now. When sample load increases, we will need to use a fast efficient system to get data to engineers, programs, and external customers. The "data package" system used for single-shell tank characterization is not fast enough.
- 5. We need to develop back-ups to current thermal analysis staff.
- 6. We need to have a low-temperature drying method for adiabatic samples that is controlled well enough to allay any suspicion that the samples lose their chemical energy before the actual adiabatic measurement.

No formal actions were taken or assigned at this meeting. These minutes are issued as a way to raise general awareness that needs for direct measurement of waste sample energetics has the potential for dramatic growth. The laboratory should be prepared with respect to knowledge of methods, manpower, and equipment.

Table A-1. Summary of Contents and Status of Ferrocyanide Tanks.

Tank	Total waste volume (1,000 gal)	FeCN ^b (1,000 g mol)	Heat load (1,000 Btu/h) ^c		imum np. (°F)	Status of tanks
BX-102	96	< 1	< 10	17	63	S; AL
BX-106	45	< 1	< 10	17	62	NS; Sound
BX-110	199	< 1	< 10	18	64	S; AL
BX-111	230	< 1	< 10	20	68	NS; AL
BY-101	387	< 1	8.2	24	76	S; Sound
BY-103	400	66	8.6	28	82	NS; AL
BY-104	406	83	5.5 - 11.0 ⁴	54 46 ^f	129 115	S; Sound
BY-105	503	36	4.0 - 8.0 ^d	46 50	115 122	NS; AL
BY-106	642	70	5.5 - 11.0 ^d	54	130	NS; AL
BY-107	266	42	14.5	35	95	S; AL
BY-108	228	58	4.4 - 8.8 ^d	43	110	S; AL
BY-110	398	71	4.0 - 8.0 ^d	49 43 ^f	120 109	S; Sound
BY-111	459	6	2.4 - 4.8 ^d	30	86	S; Sound
BY-112	291	2	< 10	28	82	S; Sound
C-108	66	25	< 10	22	72	S; Sound
C-109	66	30	3.5 - 7.0 ^d	23 26 ^r	74 78	S; Sound
C-111	57	33	< 10	22	71	S; AL
C-112	109	31	< 10	29 29	84 85	S; Sound
T-101	133	< 1	< 10	21	69	NS; AL
T-107	180	5	< 10	19	66	NS; AL
TX-118	347	< 3	4.9	26	78	S; Sound

Table A-1. Summary of Contents and Status of Ferrocyanide Tanks.

Tank	Total waste volume (1,000 gal)	FeCN ^b (1,000 g mol)	Heat load (1,000 Btu/h) ^c	Maximu temp. (°C) (°)	Status of tanks
TY-101	118	23	< 10	18 6	5 S; AL
TY-103	162	28	< 10	19 6	6 S; AL
TY-104	46	12	< 10	17 6	3 S; AL
Totals	5,834,000 gal	624K g-mol.			

^{*}Based on information contained in monthly reports (WHC-EP-0182-XX) (Hanlon 1993); temperature data as of March 1993.

^bInventories from Borsheim and Simpson, 1991.

^{&#}x27;Heat load values are conservatively high; new values will be calculated.

⁴New heat load data as of September 1992, showing low and high end of range based upon variances in thermal conductivities for waste and soil.

^{&#}x27;S - Interim Stabilized Tank; NS - Not Stabilized; AL - Assumed Leaker Tank; Sound - Non-Leaking Tank.

Temperatures recorded for new thermocouple trees installed in September 1992.

APPENDIX D

QUALITY ASSURANCE DOCUMENTATION SUMMARY

This page intentionally left blank.

Table I-1: SST Core 47, PNL-ACL Sample Numbers

	C47 COMPOSITE	CORE47-18	CORE47-1C	CORE47-1D	
ACID	93-01358-A1				Acid Digest, Sample (ICP & Sb)
DIGESTION	93-01358-A2				Acid Digest. Duplicate
	93-01358-A3				Methods Blank
	93-01358-A4				Matrix Spike
	93-01358-A5				Spike Control
ACID	93-01358-B1				Acid Digest. Sample (AA) As & S
DIGESTION	93-01358-82				Acid Digest, Duplicate
(AA)	93-01358-B3				Methods Blank
	93-01358-B4				Matrix Spike
	93-01358-B5			}	Spike Control
WATER	93-01358-C1	93-01355-C1	93-01356-C1	93-01357-C1	Water Leach Sample
LEACH	93-01358-C2	93-01355-C2	93-01356-C2	93-01357-C2	Water Leach Duplicate
	93-01358-C3	93-01355-C3		33 31331 32	Methods Blank
	i	93-01355-C4			Matrix Spike
		93-01355-C5			Spike Control
MERCURY	93-01358-D1				Mercury Sample
	93-01358-D2	i			Mercury Duplicate
	93-01358-D3				Methods Blank
	93-01358-D4				Mercury Spike
	93-01358-D5				Mercury Standard
SEMI-VOA	93-01358-E1				Total Control
	93-01358-E2				Semi-VOA Sample Semi-VOA Duplicate
	93-01358-E3	•			Matrix Spike
	93-01358-E4				Matrix Spike Duplicate
BOX	Insufficient	********************************	^1000000001000000000000000000000000000		
	Sample				EOX Sample
	Available				EOX Duplicate Methods Blank
	1			1	Matrix Spike
FUSION	93-01358-H1	93-01355-H1	50 01050 H4		
DISSOLUTION	93-01358-H2	93-01355-H2	93-01356-H1 93-01356-H2	93-01357-H1 93-01357-H2	Fusion Sample (ICP & Radchem)
	93-01358-H3	93-01355-H3	93-01330-HZ	93-01357-42	Fusion Duplicate
CARBON	93-01358-J1	*************			Methods Blank
CANBOIT	93-01358-J1 93-01358-J2	93-01355-J1	93-01356-J1	93-01357-J1	Carbon Sample (TOC/TIC/TC)
	93-01358-J2 93-01358-J3	93-01355-J2	93-01356-J2	93-01357-J2	Carbon Duplicate
	33-01338-03	93-01355-J4			Methods Blank
14.55		งิชรัชราชอดภาราร รายอังสามารถสามารถสุดสุดสุดสุดสุดสุดสุดสุดสุดสุดสุดสุดสุดส			Spiked Sample
WT%	93-01358-K1	93-01355-K1	93-01356-K1	93-01357-K1	Wt. % Solids sample
SOLIDS	93-01358-K2	93-01355-K2	93-01356-K2	93-01357-K2	Wt. % Solids Duplicate
DSC/TGA	93-01358	93-03117	93-03118	93-03119	
ARTICLE SIZE	93-01352			* * * * * * * * * * * * * * * * * * *	
RHEOLOGY	93-01353	ĺ			

Water Leach Sample for Composite (ICP, IC/CN, NH3, Cr(VI), TOC, C14, pH, H-3, GEA, Total Alpha, Total Beta) Water Leach Sample for Quarter Segments (IC/CN, pH)

Radiochemical Analyses (Total Alpha, Total Beta, GEA, U, Tc, Sr/Y, Se, Alpha Pu, Am, Pu/U isotopics

Table I-2: SST Core 48, PNL-ACL Sample Numbers

	C48 COMPOSITE	CORE 48-1C	CORE 48-1D	
ACID	93-01363-A1		93-01361-A1	Acid Digest. Sample (ICP & Sb)
DIGESTION	93-01363-A2		93-01361-A2	Acid Digest, Duplicate
ţ			93-01361-A3	Methods Blank
	93-01363-A4			Matrix Spike
nentoscoccocococococona anaeriditaca	dr. 0.0.0.0.0.0.0.0.0.0.0.0.0.0.0.0.0.0.0	000000000000000000000000000000000000000		Spike Control
ACID	93-01363-81			Acid Digest. Sample (AA) As & Se
DIGESTION	93-01363-B2			Acid Digest. Duplicate
(AA)]			Methods Blank
	93-01363-B4			Matrix Spike
			60.000000000000000000000000000000000000	Spike Control
WATER	93-01363-C1	93-01360-C1	93-01361-C1	Water Leach Sample
LEACH	93-01363-C2	93-01360-C2	93-01361-C2	Water Leach Duplicate
	1		93-01361-C3	Methods Blank
	İ		93-01361-C4	Matrix Spike
			93-01361-C5	Spike Control
MERCURY	93-01363-D1		***************************************	Mercury Sample
	93-01363-D2		•	Mercury Duplicate
j				Methods Blank
]	93-01363-D4			Mercury Spike
				Mercury Standard
SEMI-VOA	Insufficient			Semi-VOA Sample
	Sample			Semi-VOA Duplicate
	Available			Matrix Spike
				Matrix Spike Duplicate
ECX	Insufficient		000000000000000000000000000000000000000	EOX Sample
ĵ	Sample			EOX Duplicate
	Available			Methods Blank
	[Matrix Spike
FUSION	93-01363-H1	93-01360-H1	93-01361-H1	Fusion Sample (ICP & Radchem)
DISSOLUTION	93-01363-H2	93-01360-H2	93-01361-H2	Fusion Duplicate
ļ			93-01361-H3	Methods Blank
CARBON	93-01363-J1	93-01360-J1	93-01361 <i>-</i> J1	Carbon Sample (TOC/TIC/TC)
	93-01363-J2	93-01360-J2	93-01361-J2	Carbon Duplicate
			93-01361-J3	Methods Blank
li li	Ì	Ì	93-01361-J4	Spiked Sample
WT%	93-01363-K1	93-01360-K1	93-01361-K1	Wt. % Solids sample
SOLIDS	93-01363-K2	93-01360-K2	93-01361-K2	Wt. % Solids Duplicate
DSC/TGA	93-01363	93-03120	93-03121	
23071GA	30-01000	33-03 (20	9J-VJ &	

Water Leach Sample for Composite (ICP, IC/CN, NH3, Cr(VI), TOC, C14, pH, H-3, GEA)

Water Leach Sample for Quarter Segments (IC/CN, pH)

Radiochemical Analyses (Total Alpha, Total Beta, GEA, U, Tc, Sr/Y, Se, Alpha Pu, Am, Pu/U isotopics

Table I-3: SST Core 49, PNL-ACL Sample Numbers

	C49 COMPOSITE	CORE49-1B	CORE49-1C	CORE49-1D	
ACID	93-01371-A1			93-01367-A1	Acid Digest. Sample (ICP & Sb)
DIGESTION	93-01371-A2			93-01367-A2	Acid Digest. Duplicate
	93-01371-A3			93-01367-A3	Methods Blank
	93-01371-A4				Matrix Spike
	93-01371-A5				Spike Control
ACID	93-01371-81		****************		Acid Digest. Sample (AA) As &
DIGESTION	93-01371-82				Acid Digest. Duplicate
(AA)	93-01371-83				Methods Blank
	93-01371-84				Matrix Spike
	93-01371-B5				Spike Control
WATER	93-01371-C1	93-01365-C1	93-01366-C1	93-01367-C1	Water Leach Sample
LEACH	93-01371-C2	93-01365-C2	93-01366-C2	93-01367-C2	Water Leach Duplicate
					Methods Blank
					Matrix Spike
					Spike Control
MERCURY	93-01371-D1				Mercury Sample
	93-01371-D2	ł			Mercury Duplicate
			•		Methods Blank
	93-01371-D4				Mercury Spike
				**	Mercury Standard
SEMI-VOA	93-01371-E1				Semi-VOA Sample
	93-01371-E2				Semi-VOA Duplicate
:	93-01371-E3				Matrix Spike
	93-01371-E4				Matrix Spike Duplicate
EOX	93-01371-F1				EOX Sample
	93-01371-F2				EOX Duplicate
	93-01371-F3				Methods Blank
	93-01371-F4	•			Matrix Spike
FUSION	93-01371-H1	93-01365-H1	93-01366-H1	93-01367-H1	Fusion Sample (ICP & Radchem
DISSOLUTION	93-01371-H2	93-01365-H2	93-01366-H2	93-01367-H2	Fusion Duplicate
			00 0,000 1,12	93-01367-H3	Methods Blank
CARBON	02 01271 11	93-01365-J1	93-01366-J1	93-01367 <i>-</i> J1	# 1000000000000000000000000000000000000
CARBON	93-01371-J1 93-01371-J2	93-01365-J1 93-01365-J2	93-01366-J1 93-01366-J2	93-01367-J1 93-01367-J2	Carbon Sample (TOC/TIC/TC) Carbon Duplicate
	93-01371-J2 93-01371-J3	30-01303-02	33-01300-02	93-01367-32	Methods Blank
	93-013/1-33	93-01365-J4		93-01367-03	Spiked Sample
14FTP*	00 01074 74	00000000000000000000000000000000000000	00.01000.41	00.01007.65	**************************************
WT%	93-01371-K1	93:01365-K1	93-01366-K1	93-01367-K1	Wt. % Solids sample
SOLIDS	93-01371-K2	93-01365-K2	93-01366-K2	93-01367-K2	Wt. % Solids Duplicate
DSC/TGA	93-01371	93-03122	93-03123	93-03124	

Water Leach Sample for Composite (ICP, IC/CN, NH3, Cr(VI), TOC, C14, pH, H-3, GEA)

Water Leach Sample for Quarter Segments (IC/CN, pH)

Radiochemical Analyses (Total Alpha, Total Beta, GEA, U, Tc, Sr/Y, Se, Alpha Pu, Am, Pu/U Isotopics

Table I-4: SST C-109, Liquid Composite and Hot Cell Blank, PNL-ACL Sample Numbers

	TANK C-109 L	IQUID COMP	OSITE & HO	CELL BL	ANK
		E PREPARAT			
					•
<u> </u>	Liquid Composite	DIW DIL CHECK	Hot Cell Blank	DIW Blank	
ACID	93-01354-A1	93-03290	1		Acid Digestion Sample (ICP)
DIGESTION	93-01354-A2]	Acid Digestion Duplicate
	93-01354-A3			i	Methods Blank
	93-01354-A4				Spike
	93-01354-A5		***************************************		Spike Control
WATER	93-01354-C1	93-03290			Water Leach Sample
LEACH	93-01354-C2		•		Water Leach Duplicate
	93-01354-C3			1	Methods Blank
	93-01354-C4				Spike
	93-01354-C5				Spike Control
MERCURY			93-01327-D1	93-01372-0	Sample for Hg
			93-01327-D2	00-01072-0	Duplicate
			93-01327-D3		Methods Blank
SEMI - VOA	93-01354-E1	93-03290	93-01327-E1	62 01272 E	Sample S-VOA
52 75	93-01354-E2	30-03230	93-01327-E2	93-013/2-2	
	93-01354-E3		93-01327-62	İ	Duplicate S-VOA Matrix Spike
ļ	93-01354-E4				Matrix Spike Dup.
	30-01004-24		93-01327-E3		Methods Blank
тох			93-01327-F1	93-01372-F	Sample for TOX
			93-01327-F2		Duplicate
***********			93-01327-F3		Methods Blank
DIRECT	93-01354-G1	93-03290	93-01327-G1	93-01372-G	Direct Sample *
	93-01354-G2		93-01327-G2		Duplicate
	93-01354-G3	· •	93-01327-G3	000000000000000000000000000000000000000	Methods Blank
DIRECT	93-01354-N1	93-03290	93-01327-N1	93-01372-N	Direct Filtered Sample **
FILTERED	93-01354-N2		93-01327-N2		Direct Duplicate
ļ	93-01354-N3		93-01327-N3		Methods Blank
	93-01354-N4		•		Spike
	93-01354-N5	•			Spike Control

Water Leach Analyses (ICP, IC, CN-, TOC, NH3)

Pu/U MS, Alpha Pu & Am, C-14) Hot Cell Blank Analyses (IC & TOC)

^{*} Liquid Composite Analysis (CN) Hot Cell Blank Analyses (Total Alpha, Total Beta, GEA, ICP, AA)

^{**} Liquid Composite Analyses (pH, Hg, OH-, Total Alpha, Total Beta, GEA, U, Tc, Sr, I, H-3,

associated with the sludge. Some Liquid Composite and 325-A Hot-Cell Blank samples were prepared for analyses by the ACL technical groups outside of the cells due to their low radioactivity levels and a concern for potential incell contamination problems.

Table 1-2 lists the procedures that were employed to prepare Tank C-109 samples for analyses or to conduct a limited number of in-cell analytical determinations:

Table 1-2: Shielded Analytical Laboratory Procedure List for Tank C-109

PNL Procedure Number	Procedure Title
PNL-ALO-101, Rev. 1	Acid Digestion for Metals Analysis
PNL-ALO-102, Rev. 0	Fusion of Hanford Tank Waste Solids
PNL-ALO-103, Rev. 1	Water Leach of Sludges, Soils, and Other Solid Samples
PNL-ALO-213, Rev. 0	Mercury in Water, Solids, and Sludges by Manual Cold Vapor Technique
PNL-ALO-120, Rev. 0	Extraction of Single Shell Tank Samples for the Analysis of Semi-Volatile Organic Compounds
PNL-ALO-225, Rev. 0	Measurement of pH in Aqueous Solution
PNL-ALO-320, Rev. 0	Method for Extractable Organic Halides (EOX) in Solids
PNL-ALO-504, Rev. 1	Percent Solids Determination of Hanford Tank Waste Sludges
PNL-7.40.42, Rev. 0	Determination of Carbon-14 in Radioactive Liquids, Soils, and Sludges

ŧ

Low sample recovery required that a reduced work scope be performed on Core 47 and Core 48 composite material. The LOI (9258244) from Hanford Analytical Services Management (HASM) resulted in test instructions that prioritized the order in which samples were processed. Acid digestions for ICP and GFAA, fusions, water leaches, and mercury preparations were conducted on all three cores. Subsamples were aliquoted from Cores 47 and 49 for SVOA. Samples from Core 49 were weighed for EOX.

- 1) Insufficient sample was available to conduct the analyses per procedure while maintaining the level of quality control requested.
- 2) Sample weights and/or final volumes were reduced to facilitate waste minimization.
- Sample weights and/or final volumes were altered to increase the concentration of certain analytes of interest. This was done to meet the procedural concentration ranges needed to perform the analyses.

The following table lists the sample preparatory procedure deviations performed during the processing of Tank C-109:

Table 1-3: Tank C-109 Sample Preparation Procedure Deviations

	TE 1 3. Tank		rreparation		
Sample ID	ACL Number	Sample Size Deviation	Sample Volume Deviation	Reagent Deviation	Observed Effect
47/49-LC	93-01354-A	No	Yes	No	None
47-CC	93-01358-A	Yes	Yes	No	None
48-1D	93-01361-A	Yes	Yes	No	None
48-CC	93-01363-A	Yes	Yes	No	None
49-10	93-01367-A	Yes	Yes	No	None
49-CC	93-01371-A	Yes	Yes	No	None
47-CC	93-01358-B	Yes	Yes	No	None
48-CC	93-01363-8	Yes	Yes	No	None
49-CC	93-01371-B	Yes	Yes	No	None
47-18	93-01355-C	Yes	Yes	No	None
47-1C	93-01356-C	Yes	Yes	No	None
47-10	93-01357-C	Yes	Yes	No	None '
47-CC	93-01358-C	No No	Yes	No	None
48-1C	93-01360-C	Yes	Yes	No	None
48-1D	93-01361-C	Yes	Yes	No	None
48-CC	93-01363-C	Na	Yes	No	None
49-18	93-01365-C	Yes	Yes	No	None
49-1C	93-01366-C	Yes	Yes	No	None
49-1D	93-01367-C	Yes	Yes	No	None
49-CC	93-01371-C	No	Yes	No	None

Table 1-3: Tank C-109 Sample Preparation Procedure Deviations

	ic i o. Tank				
47-CC	93-01358-0	No	Yes	No	None
48-CC	93-01363-0	No	Yes	No	None
49-CC	93-01371-0	No	Yes	No	None
47-18	93-01355-н	Ма	Yes	No	None
47-1C	93-01356-Н	No	Yes	Na	None
47-1D	93-01357-н	No	Yes	No	None
47-CC	93-01358-Н	No	Yes	No	None
48-1C	93-01360-Н	No	Yes	No	None
48-10	93-01361-H	No	Yes	No	None
48-CC	93-01363-Н	No	Yes	No	None
49-18	93-01365-Н	No	Yes	No	None
49-1C	93-01366-H	No	Yes	No	None
49-1D	93-01367-Н	No	Yes	No	None
49-CC	93-01371-Н	No	Yes	No	None
47-1B	93-01355-K	Yes	No	Мо	None
47-1C	93-01356-K	Yes	No	No	None
47-10	93-01357-K	Yes	No	No	None
47-CC	93-01358-K	Yes	No	No	None
48-1C	93-01360-K	Yes	Na	No	None
48-1D	93-01361-K	Yes	No	No	None
48-CC	93-01363-K	Yes	No	No	None
49-18	93-01365-K	Yes	No	No	None
49-1C	93-01366-K	Yes	No	No	None
49-10	93-01367-K	Yes	No	No	None
49-CC	93-01371-K	Yes	No	No	None

It should be noted that all deviations performed were minor changes to parameters such as sample size and final volume, and that these changes did not alter the actual chemistry involved. Sample sizes and final volumes for all sample preparations are documented in the project records and are included in Appendix C.

SST Cores 47 and 49, Differential Scanning Calorimetric Data

Table 1-4a:

			Tran	is noite			Trac	nsition #2			Transition #3			
Sample	Run	Range (C)	Onset (C)	Peak (C)	Enthalpy (J/g)	Range (C)	Onset (C)	Peak (C)	Enthalpy (J/g)	Range (C)	Onset (C)	Peak (C)	Enthalpy -{J/g}	
													12: 81	
Core 47 1B	1]]	33-150	83	93	538	190-336	259	280	1594		NONE OB	SERVED		
	2	35-150	57	85	401	190-338	259	280	1518		NONE OF	SERVED		
Core 47 1C	1	33-144	50	8.5	429	167-314	217	282	605	380-455	386	404	92	
	2	35-144	5.6	92	421	167-318	217	284	614	380-461	396	418	52	
Care 47 1D	,	34-146	54	8 1	816	190-358	213	273	548	369-441	375	391	21	
	2	34-154	64	86	919	194-369	236	274	467	1	NONE OBSERVED			
Core 47 Comp.		34-150	56	93	798	166-330	216	284	1106		NONE OB	NONE OBSERVED		
	2	34-150	5.5	02	772	159-322	216	283	1052	1	NONE OB	SERVED		

Core 49 1B		35-115	47	85	429	193-373	270	285	2393		NONE OB	SERVEN		
	2	33-109	33	78	306	197-341	270	285	1982		NONE OB			
Core 49 1C	,[]	35-197	110	124	726	197-316	248	280	530		NONE OB	SERVED		
	2	33-153	33	105	587	167-311	237	285	599		NONE OB			
Core 49 1D	١ ا	34-150	8.5	9.9	691	152-324	220	276	324	379-474	390	408	42	
	2	35-166	77	113	733	186-315	230	273	286	379-483	397	420	5.3	
Core 49 Comp.	1	34-192	110	122	1307	194-325	246	265	1216		NONE OB	SERVED		
	2	34-168	88	119	620	190-329	240	287	628		NONE OB	NONE OBSERVED		

APP D-8

SST Cores 48, Differential Scanning Calorimetric Data

Table 1-4b:

		Transition #1				Transition #2				
Sample	Run	Range (C)	Onset (C)	Peak (C)	Enthalpy (J/g)	Range (C)	Onset (C)	Peak (C)	Enthalpy (J/g)	
Core 48 1D	1 2	34-196 34-166	112 95	124 	1121 947	196-255 185-237	198 198	212 209	7 23	

			Tran	sition #3		Transition #4						
Sample	Run	Range (C)	Onset (C)	Peak (C)	Enthalpy (J/g)	Range (C)	Onset (C)	Peak (C)	Enthalpy (J/g)			
Core 48 1D	1 1 2	249-336 252-338	270 273	286 290	-26 -28	336-431 338-419	358 359	384 388	24 38			

Negative enthalpies indicate exothermic behavior.

Table 1-5: SST Cores 47, 48, and 49, Scanning Thermogravimetric Data

		Tran	nsition #1	Tran	sition #2	Trai	nsition #3
Sample	Run	Range (C)	Mass Loss (%)	Range (C)	Mass Loss (%)	Range (C)	Mass Loss
Core 47 18	1	31-150	10.0	150-336	18.0	336-500	2.5
	2	31-150	10.4	150-336	17.8	336-500	3.5 3.1
Core 47 1C	1	31-150	18.1	150-336	17.8	336-500	3.7
٠	2	31-150	18.0	150-336	17.4	336-500	3.7
Core 47 †D `	1	31-150	19.7	150-370	7.0	370-500	1.5
	2	31-150	19.7	150-370	6.5	370-500	1.8
Care 47 Comp.	1	31-150	15.6	150-336	14.7	370-500	3,4
	2	31-150	14.0	150-336	15,1	370-500	4.0

Core 48 1D	1	31-180	46.4	180-425	3.2	425-500	-0.2
	2	31-180	43.8	180-425	3.1	425-500	-0.2
Core 49 1B	1	31-180	2.4	180-336	26.3	336-500	4.3
	2	31-180	6.0	180-336	25.3	336-500	3.8
Core 49 1C	1	31-180	28.6	180-336	14.3	336-500	2.7
	2	31-180	30.6	180-336	14.1	336-500	2.9
Care 49 1D	1	31-180	29.6	180-350	9.4	350-500	1.1
	2	31-180	29.0	180-350	9.7	350-500	1.1
Core 49 Comp.	1	31-180	27.5	180-336	15.1	336-500	3.5
	2	31-180	25.6	180-336	16.4	336-500	3.9

PHYSICAL PROPERTIES

The physical properties which were measured on this core included weight percent solids, pH, and particle size distribution. The wt% total solids were obtained for each of the quarter segments and the core composites. The total solids analyses were performed according to PNL technical procedure PNL-ALO-504, "Weight Percent Solids." This analysis is a gravimetric determination of the wt% solids as measured by the loss of mass in the sample after being held in a drying oven at 105°C for 24 hours. The wt% water is calculated by subtracting the weight percent solids from 100.

The wt% water values are given in Table 1-6. The wt% waters of the core composite from Core 48 was significantly higher than the average of the quarter segments from the Core 48. The core composite for Core 48 was the subsample originally taken for VOA. This sample was taken immediately after extrusion and prior to weighing the sample and taking pictures. This difference in handling may account for the higher wt% water values measured in these samples. The wt% waters measured on the other core composites (Cores 47 and 49) are lower than was observed in the average of the quarter segment samples. This is due to increased handling of the core composite material in the hot-cell environment.

Table 1-6: Cores 47, 48, and 49, Weight Percent Solids

Core	Sample	ACL Number	VtX Vater
47	1B	93-01355	19.3
	1C	93-01356	28.4
	10	93-01357	39.4
	Composite	93-01358	21.5
48	1C	93-01360	52.8
	10	93-01361	51.6
	Composite	93-01363	57.7
49	18	93-01365	19.6
···	1C	93-01366	38.3
	10	93-01367	39.6
	Composite	93-01371	27.8

Table 2-1a: SST Core 47, ICP Core Composite Fusion

ICP ANALYSIS REPORT -- KOH/Ni Fusions

PROJECT: SST PROCEDURE: PNL-ALG-211 Analyzed Date: 01/06/93 M&TE: WA55672

Samp Log#: 9 Dilution: ug/g Factor ICP Run #	H1 73-01358 9 1.00 3979.4 399	H2 73-01358 1.00 4379.4 400						ank H3 1-01358 1.00 4169.8 398			
	Sample ug/g	Dupi. ug/g	RPO	20% Flag	Sample ug/g	Dupl. ug/g	20% RPO Flag	Blank ug/g	Blk-Dup ug/g	**"Estima OL ug/mL	dr dr
Aglas Baecococopekaignoadibenubeirehi	9/9	**************************************	3.3	Flag	ug/g	ug/g	RPO Flag	49/9	∪ q / q	0.0078 0.1829 0.1014 0.0596 0.0080 0.0032 0.0016 0.0075 0.1019 0.1865 0.0113 0.0088 0.0053 0.0100 0.3173 0.0124 0.0074 0.0003 0.01128 0.0587 0.0496 0.0231 0.0821 0.0821 0.0821 0.0821 0.0549 0.0549 0.0549	Ug/mL
TL U V Zn Zr P	<pre><0L 8,745 <0t 329 <0L 20,152</pre>	<0L 9,609 <0L 372 <0L	9.4 12.3 2.8					<0L <0L <0L (34) <0L <0L		0.5492 0.5376 0.0090 0.0041 0.0076 0.6133	1.3308 1.7920 0.0301 0.0136 0.0253 2.0442

Note: 1) Values reliable to 2 1/2 significant digits. Bracketed results () are qualitative.
2) Blank is reported in ug/g "equivalence" to indicate blank effect on sample.
3) Sample results have not been adjusted for "blank" contribution.
4) At 50-100 times the D.L., precision is estimated at +/-10% and accuracy at +/-15%.
5) Sample OL (ug/g) = (OL in ug/mL) * (ug/g factor)
6) Off-line IEC: Results within 50% OL potentially bias high.
7) 20% "** flag: RPD > 20% and both sample results > 3*OL.

01/07/93

Data, including calibration/QC, archived File ICP-325-601- 01/06/93

Table 2-1b: SST Core 48 and 49, ICP Core Composite Fusion

ICP ANALYSIS REPORT -- KOH/NI Fusions

PROJECT: SST

PROCEDURE: PHL-ALO-211

Analyzed Oate: 01/06/93 H&TE: WA55672

Samp Log#: 9: Dilution: ug/g factor ICP Run #	1.00 2994.0 401	1.00 2750.3 402			H1 3-01371 1.00 2446.3 403	1.00 2190.1 404			Blank analy 93-01358	zed with	***Estime	ated***	
	Sample ug/g	Oupl. ug/g	RPD	20% Flag	ug/g	0upł. ug/g	RPD	20% Flag	Blank ug/g	Blk-Oup ug/g	OL ug/mL	at ug/mt	
Nd Ni Pb Re Rh Sb Sc Si Sr Te Th Ti U V Zn	7,280 7,280 90L 90L 90L 16,782 90L 268 (50) 90L 23,818 9/A 90L 90L 90L 90L 90L 90L 90L 90L 90L 90L	**COL	4.8 16.2 13.7	•	0L 119,500 0L 0L 0L 0L 14,476 0L 215 (61) 0L 225 (61) 0L 334 0L 334 0L 0L 2,244 167 0L 0L 2,244 167 0L 0L	**OL** **133,679** **OL** **(41)* **OL** ** **OL** **OL** **OL** **OL** **OL** **OL** **OL** **OL** **OL**	5.2 1.6 3.6 3.8				0.0078 0.1829 0.1014 0.0596 0.0030 0.0032 0.0016 0.0075 0.1019 0.1865 0.0113 0.0053 0.0100 0.3173 0.0124 0.0074 0.0031 0.01010 0.0128 0.0587 0.0496 0.0231 0.0350 0.0101 0.0128 0.0587 0.0587 0.0587 0.0587 0.0587 0.0587 0.0587 0.0587 0.0587 0.0587 0.0587 0.0587	0.0261 0.6096 0.3379 0.1987 0.0267 0.0107 0.0054 0.0250 0.3396 0.6217 0.0378 0.0378 0.0176 0.0334 1.0576 0.0414 0.0246 0.0009 0.0034 0.0427 0.1653 0.0768 0.2738 0.1167 0.1584 0.2738 0.1167 0.1584 0.1584 0.1584 0.1683 0.1167 0.1830 0.1830 0.013 0.1830 0.013 0.1830 0.0179 1.8308 1.7920 0.0331 0.0136	- Production of the Company of the
Zr P	<0L 22,210	<0L 18,154	20.1	•	<0L 17,745	<0L 11,435	43.3				0.0076 0.6133	0.0253 2.0442	

- Note: 1) Values reliable to 2 1/Z significant digits. Bracketed results () are qualitative.
 2) Blank is reported in ug/g "equivalence" to indicate blank effect on sample.
 3) Sample results have not been adjusted for "blank" contribution.
 4) At 50-100 times the D.L., precision is estimated at +/-10% and accuracy at +/-15%.
 5) Sample DL (ug/g) = (DL in ug/mL) * (ug/g factor)
 6) Off-line IEC: Results within 50% OL potentially bias high.
 7) 20% **** flag: RPD > 20% and both sample results > 3*QL.

Data, including calibration/QC, archived File ICP-325-601- 01/06/93

01/07/93

Table 2-Ic: SST Core 47 and 48, ICP Quarter Segments Fusion

ICP ANALYSIS REPORT -- KOH/Ni Fusions

PROJECT: SST PROCEDURE: PNL-ALO-211 Analyzed Date: 01/08/93

H&TE: WA55672

Samp Log#: 9 Dilution: ug/g Factor ICP Run #	1.00	H2 93-01355 1.00 2964.5 414		9	#1 3-01360 1.00 2312.7 420			•	8 lank 93-01355 1.00 2939.7 412			
ter kari w	713	717			720	761			412		***Estima	****
	Sample	Oupl.		20%	Sample	Dupl.		20%	Blank	Blk-Oup	OL	OL.
	ug/g	ug/g	RPD	Flag	ug/g	ug/g	RPD	flag		ug/g	ug/mt	ug/mL
				• • • •				•			••••••	
Ag	40t	<0L	11 0		<0L	<0L	4.2		<0L		0.0078	0.0261
AL	123,876 <0L	139,344 <0L	11.8		7,440	7,135 <0L	4.2		<0L		0.1829	0.6096
AS B	<0L	<0L			<0L <0L	<0L			<0L		0.1014	0.3379
=	(84)				84	(75)			<0L		0.0596	0.1987
Ba	(04, <0L	, 100 <0L			<0L	(75) <0L			<0L		0.0080	0.0267
9e Ca	10,681		4.5		30, 182		6.2		<0↓ 262		0.0032	0.0107
Cd			4.3		30,102 <0L	•	0.2				0.0016	0.0054
	<0L	<0↓ <0↓				<0L			<0L		0.0075	0.0250
Ce	<0L				<0L				<0L		0.1019	0.3396
Co	<0L	<0L	3.1		<0L 478	<0L	/2 1		<0L		0.1865	0.6217
Cr	204	211					42.1		<0L		0.0113	0.0378
Cu	199	_	2.8		152				(29)		0.0088	0.0294
Dy	<0L	<0L	40 E		40L		/	_	<0£		0.0053	0.0176
f e	82,536		60.5	-	22,752	-	27.0	•	610		0.0100	0.0334
K	N/A	N/A			N/A				H/A		0.3173	1.0576
La . :	(111)				<0L	<0L			<0L		0.0124	0.0414
Li	40L	<0L			<0∟ 732				<0L		0.0074	0.0246
Mg	1,200 505	615 425			732 238		8.5		65		0.0003	0.0009
Mn	<0L		17.2				14.0		121		0.0010	0.0034
Mo Na	49,692	(48) 52,421	5.3		37) 138,336		38.8		₹0 L		0.0128	0.0427
Nd	47,672 40L	J2,421 ≺OL	٠.٠		10°, 20°,		30.0	-	1,164		0.0587	0.1957
Ni Ni	N/A	N/A			H/A				<0L		0.0496	0.1653
Pb	5,530	4,568	19.1		(635				N/A		0.0231	0.0768
Re	7,330 <0L	4,500 <0L	17.1		(035 40k	, (4/3) <ol< td=""><td></td><td></td><td><0L <0L</td><td></td><td>0.0831 0.0173</td><td>0 ::771</td></ol<>			<0L <0L		0.0831 0.0173	0 ::771
Rh	₹0 L	<0L			<0L	<0L			₹0 L		0.0173	. 0576
Ru	<0L	√0L			40L	₹0 L			√ 0L		0.0350	0.2738
Sb	<0L	≺OL			<0L	<0L			√0L		0.0330	0.116 <i>7</i> 0.1584
Se	< 0L	<0L			<0L	√0 Ł			.0£		0.1515	
Sí	18,466	18,968	2.7		3,420		37.1	*	<0L		0.0549	0.5050
\$r	169	166	1.9		657		18 1		₹ 0L		0.0004	0.1830
Te	<0L	<0L	,		<0L	<0L	10.1		<0L		0.0004	0.0013 0.3168
Th	حەل	<0L			<0L	<0L			<0L			
Ţí	424	392	7.7		<0L	69					0.0734	0.2448
Ťĺ	<0L	<0L	,		<0L	<0L			<0↓ <0↓		0.0054	0.0179
ΰ`	11,529		4.3		18,058		16.0				0.5492	1.8308
v	,32, ≪0L	(OL	7.3		10,03G 40L	13,381 ≪0L	10.0		₹0 L		0.5376	1.7920
Žn	344	341	0.8		398		13.9		<0∟ 54		0.0090	0.0301
21	(53)		5.5		<0L		13.7		_		0.0041	0.0136
P'	(7,366)				26,246		25 7		- ۱۵۰		0.0076	0.0253
F	(1,500)	, 1,007			40,240	40,239	43.1	-	<0L		0.6133	2.0442

Note: 1) Values reliable to 2 1/2 significant digits. Bracketed results () are qualitative.

2) Blank is reported in ug/g "equivalence" to indicate blank effect on sample.

3) Sample results have not been adjusted for "blank" contribution.

4) At 50-100 times the 0.L., precision is estimated at +/-10% and accuracy at +/-15%.

5) Sample DL (ug/g) = (DL in ug/mL) = (ug/g factor)

6) Off-line IEC: Results within 50% DL potentially bias high.

7) 20% *** flag: RPD > 20% and both sample results > 3*QL.

Data, including calibration/QC, archived File ICP-325-601- 01/08/93

01/11/93

Table 2-1d: SST Core 47, ICP Quarter Segments Fusion

ICP ANALYSIS REPORT -- KOH/NI fusions

PROJECT: SST PROCEDURE: PNL-ALG-211

Analyzed Date: 01/08/93 H&TE: WAS5672

Samp Log#: 9 Dilution: ug/g factor ICP Run #	1.00 2267.2	н2 93-01356 1.00 2451.0 416		9	н1 93-01357 9 1.00 3187.3 418	H2 73-01357 1.00 2495.8 419			llank analy 3-01355 &			
	Sample	Օս թ (.		204	Samola	Duni		204	Ol sak	Blk-Oup	***Estima	
		ug/g	RPO	Flag	Sample ug/g	ug/g	RPO	Flag	ug/g	nd/d		aL ug/mL
Ag	<0L	<0L			<0L	<0L 31,318		•	**********		0.0078	0.0261
AL	119,719		1.0		32,707	31,318	4.3				0.1829	0.6096
As	<0L	≪QL			<0L	<0L					0.1014	0.3379
8	<0L (44)	<0L (46)			<ol (77)</ol 	< 0L					0.0596	19 87
₿a	(44)	(46)			(77)	79					0.0080	0.0267
Be	<0L	<0L 17,736				<0L 27,187					0.0032	0.0107
Ca	18,359	17,736	3.5		28,779	27,187	5.7				0.0016	0.0054
Čq	<0L	<0.L <0.L			<0L	<0L					0.0075	0.0250
Ce	<0L	< 0∟			<d[< td=""><td><0L</td><td></td><td></td><td></td><td></td><td>0.1019</td><td>0.3396</td></d[<>	<0L					0.1019	0.3396
Ça	<01 242	<0L 217			< 0L	<0L					0.1865	0.6217
Çr	242	217	10.8		300	265	12.5				0.0113	0.0378
Çu	124	136 <0L 26,502	9.2		(81)	(38)					0.0088	0.0294
ŌУ	<ol< td=""><td><0L</td><td></td><td></td><td><0L</td><td><ol< td=""><td></td><td></td><td></td><td></td><td>0.0053</td><td>0.0176</td></ol<></td></ol<>	<0L			<0L	<ol< td=""><td></td><td></td><td></td><td></td><td>0.0053</td><td>0.0176</td></ol<>					0.0053	0.0176
Fe	15,380	26,502	53.1	•	17,085	13,521	23.3	•			0.0100	0.0334
ĸ	N/A	N/A			N/A	H/A					0.3173	1.0576
La	(35)	<0L			<0£	<0L					0.0124	0.0414
Li	<0L	<0L			<ol< td=""><td><0٤</td><td></td><td></td><td></td><td></td><td>0.0074</td><td>0.0246</td></ol<>	<0٤					0.0074	0.0246
Mg	363	365 252	0.5		545	577	5.8				0.0003	0.0009
Hn	197	252	24.3	•	371	207	56.6	•			0.0010	0.0034
Ho	(41)	(43)			545 371 <0L 101,673	<0₺					0.0128	0.0427
Ha	63,188	62,933	0.4		101,673	103,604	1.9				0.0587	0.1957
Иф	<dl N/A</dl 	<0L N/A			<0Ł	<0L					0.0496	0.1653
Жi	N/A	N/A			N/A	N/A 10,014					0.0231	0.0768
Pb	2,985	2,779	7.2		18,572	10,014	59.9	•			0.0831	0.2771
Re	<0L	<0L			<0L	<0L					0.0173	0.0576
Rh	<dl< td=""><td><0₺</td><td></td><td></td><td><0L</td><td>≺0↓</td><td></td><td></td><td></td><td></td><td>0.0821</td><td>0.2738</td></dl<>	< 0₺			<0L	≺0 ↓					0.0821	0.2738
Ru	<0L	<ol< td=""><td></td><td></td><td><0L</td><td><0L</td><td></td><td></td><td></td><td></td><td>0.0350</td><td>0.1167</td></ol<>			<0L	<0L					0.0350	0.1167
Şb	<0 L	<0L			<0L						0.0475	0.1584
Se	<0L	<0L			<0L	<0L 20,566 189					0.1515	0.5050
Şi	6,032	6,183		•	23,867	20,566	14.9				0.0549	0.1830
Sr	143	138	3.2		203	189	7.4				0.0004	0.0013
Te	<0L	<0L			₹UL	₹UL	·				0.0950	0.3168
<u>T</u> h	40£	<0L			<ol< td=""><td><0L</td><td></td><td></td><td></td><td></td><td>0.0734</td><td>0.2448</td></ol<>	<0L					0.0734	0.2448
Ţí	101	116	13.4		166	224 <0L	30.0			•	0.0054	0.0179
Ţι	<0L 6,615	0د 5,680 \$			<0L	<0₺					0.5492	1.8308
Ü	6,615	5,680	15.2		6.244	5,441	13.7				0.5376	1.7920
Ã	<0L 251	<0L			\U_C	\UL	_				0.0090	0.0301
Zn		276	8.6		765	232	5.5				0.0041	0.0136
Žr	«OL	<dl 12,336</dl 			<0L	<0L	_				0.0076	0.0253
Р	12,566	12,336	1.8		29,014	31,182	7.2				0.6133	2.0442

Oata, including calibration/QC, archived File ICP-325-601- 01/08/93

01/11/93

Note: 1) Values reliable to 2 1/2 significant digits. Bracketed results () are qualitative.

2) Stank is reported in ug/g "equivalence" to indicate blank effect on sample.

3) Sample results have not been adjusted for "blank" contribution.

4) At 50-100 times the 0.L., precision is estimated at +/-10% and accuracy at +/-15%.

5) Sample 0L (ug/g) = (DL in ug/mL) * (ug/g Factor)

6) Off-line IEC: Results within 50% DL potentially bias high.

7) 20% *** flag: 8PO > 20% and both sample results > 3*QL.

Table 2-le: SST Core 48 and 49, ICP Quarter Segments Fusion

ICP ANALYSIS REPORT -- KOH/Ni Fusions

PROJECT: SST

PROCEDURE: PNL-ALO-211

Analyzed Date: 01/05/93

M&TE: WA55672

Samp Log#: Dilution: ug/g Factor ICP Run #	1.00 1958.9 385	1.00 2167.7 386 Dupt.		20%	1.00 2123.7 387 Sample	2248.9 388 Dupt.		20%	Blank 93-01361 1.00 2119.4 384 8lank		***Estim DL	QL
			KFU	rtag	ug/g	ug/g	RPU	Flag	ug/g	ug/g	ug/mL	ug/mL
Ag Al As Ba Be	≪0L 9,600 ≪0L ≪0L 73 ≪0L	<0L	4.7		<pre><0L 181,114 <0L <0L (56) <0L</pre>	189,513 <0L <0L (51)			≪OL ≪OL ≪OL ≪OL ≪OL		0.0078 0.1829 0.1014 0.0596 0.0080 0.0032	0.0261 0.6096 0.3379 0.1987 0.0267 0.0107
Ca Cd Ce Co Cr	16,961 <0L <0L <0L 309	16,603 <0L <0L <0L 252		•	5,404 <0L <0L <0L 147	<0L <0L <0L			169 <0L <0L		0.0016 0.0075 0.1019 0.1865	0.0054 0.0250 0.3396 0.6217
Cu Dy Fe K	231 <0L 22,697 N/A	(49) <0L 19,379 N/A			178		15.5		<ol (44) <ol 287 N/A</ol </ol 		0.0113 0.0088 0.0053 0.0100	0.0378 0.0294 0.0176 0.0334
La Li Mg Mn	<0L <0L 590 172	<0L <0L 574	2.6 40.2		100 <0L 213 180	(72) <0L 158 245	29.8 30.3	*	<0L <0L 44 149		0.3173 0.0124 0.0074 0.0003 0.0010	1.0576 0.0414 0.0246 0.0009 0.0034
Mo Na Nd Ni	(39) 101,121 <0L N/A	102,850 <ol N/A</ol 	1.7		(55) 45,066 (123) N/A	(55) 40,977 ≪OL N/A			<0L 1,032 <0L N/A		0.0128 0.0587 0.0496 0.0231	0.0427 0.1957 0.1653 0.0768
Pb Re Rh Ru Sb	724 <0L <0L <0L <0L	662 <0↓ <0↓ <0↓	9.0		2,074 <0L <0L <0L	1,900 <0L <0L <0L	8.8	-	<0L <0L <0L <0L		0.0831 0.0173 0.0821 0.0350	0.2771 0.0576 0.2738 0.1167
Se Si Sr Te	حال	<0L <0L 1,899 459 <0L	28.5 1.7	*	<0L <0L 3,075 111		12.4 17.6		&r ≪0r ≪0r		0.0475 0.1515 0.0549 0.0004	0.1584 0.5050 0.1830 0.0013
Th Tf Tl U	<0L (31) <0L	<ol< td=""><td>3.6</td><td></td><td><0L <0L 84 <0L 8,656</td><td><0L <0L 46 <0L 7,146</td><td>58.7 19.1</td><td></td><td><0L <0L <0L</td><td></td><td>0.0950 0.0734 0.0054 0.5492</td><td>0.3168 0.2448 0.0179 1.8308</td></ol<>	3.6		<0L <0L 84 <0L 8,656	<0L <0L 46 <0L 7,146	58.7 19.1		<0L <0L <0L		0.0950 0.0734 0.0054 0.5492	0.3168 0.2448 0.0179 1.8308
V Zn Zr P	<0L 346 <0L	<0L 269 <0L 21,630	25.1		[*] <0L 664 <0L	7,148	7.0		<0L <0L 75 <0L <0L		0.5376 0.0090 0.0041 0.0076 0.6133	1.7920 0.0301 0.0136 0.0253 2.0442

Note: 1) Values reliable to 2 1/2 significant digits. Bracketed results () are qualitative.
2) Blank is reported in ug/g "equivalence" to indicate blank effect on sample.
3) Sample results have not been adjusted for "blank" contribution.
4) At 50-100 times the D.L., precision is estimated at +/-10% and accuracy at +/-15%.
5) Sample DL (ug/g) = (DL in ug/mL) * (ug/g factor)

Data, including calibration/QC, archived File ICP-325-601- 01/05/93

01/07/93

⁶⁾ Off-line IEC: Results within 50X DL potentially bias high.
7) 20% "*" flag: RPD > 20% and both sample results > 3*QL.

Table 2-1f: SST Core 49, ICP Quarter Segments Fusion

ICP ANALYSIS REPORT -- KOH/Ni Fusions

PROJECT: SST

PROCEDURE: PNL-ALO-211

Analyzed Date: 01/05/93 M&TE: WA55672

Samp Log#: 93 Dilution: ug/g Factor ICP Run #	H1 5-01366 1.00 2108.4 389 Sample	1.00 2204.3 390		20%	1.00 2228.6 391	H2 93-01367 1.00 2282.2 392			Blank analy 93-01361 & Blank	93-01365	***Estima		
	ug/g	ug/g	RPD	Flag			RPO	Flag		8lk-Dup ug/g	OL ug/mL	QĮ ug∕mL	
Ag	<0L	ØL			⟨ 0L	≪OL			*********	***********	0.0078	0.0261	
Al	97,539	94,009	3.7		73,535	68,249	7.5				0.1829	0.6096	
As	<0L	<0L			<dl< td=""><td><0L</td><td></td><td></td><td></td><td></td><td>0.1014</td><td>0.3379</td><td></td></dl<>	<0L					0.1014	0.3379	
В	<dl< td=""><td><dl< td=""><td></td><td></td><td><0L</td><td><0L</td><td></td><td></td><td></td><td></td><td>0.0596</td><td>0.1987</td><td></td></dl<></td></dl<>	<dl< td=""><td></td><td></td><td><0L</td><td><0L</td><td></td><td></td><td></td><td></td><td>0.0596</td><td>0.1987</td><td></td></dl<>			<0L	<0L					0.0596	0.1987	
Ba	(42)				(59)	(61)					0.0080	0.0267	
Be	≺0L	<0L			<0L	<0L					0.0032	0.0107	
Ça	18,864	18,248	3.3		21,336		10.5				0.0016	0.0054	
Cq	<0L	<0L			<0L	<dl< td=""><td></td><td></td><td></td><td></td><td>0.0075</td><td>0.0250</td><td></td></dl<>					0.0075	0.0250	
Ce	<0L	<0L			<0L	< 0↓					0.1019	0.3396	
Co	<0L	<0L	40.5		<0L	< 0L					0.1865	0.6217	
Cr	207		10.5		244		11.2				0.0113	0.0378	
Cu Dy	(59) <0L	164 <0L			(63)						0.0088	0.0294	
F e	4,333	4,816	10.6		.0L 13,611	<ol 17,210</ol 	23.3				0.0053	0.0176	
K	N/A	4,516 N/A	10.0		N/A	17,210 N/A	23.3	-			0.0100	0.0334	
Ĺа	. ≺0£				-7/ ∩ -<0L						0.3173	1.0576	
Ļi	<01	√OL			<0L	√0L					0.0124 0.0074	0.0414	
Mg	294		3.3		469		11.5				0.0074	0.0246 0.0009	
Mn	139	181	26.0	*	270	269	0.4				0.0003	0.0034	
Мо	(38)				(40)						0.0128	0.0034	
Na	60,864	64,841	6.3		90,234	92,536	2.5				0.0587	0.1957	
Nd	√OL	<0L			₹ 0L	<0L					0.0496	0.1653	
Ni	N/A	N/A			N/A	N/A					0.0231	0.0768	
Pb	(407)	(335)			695	762	9.2				0.0831	0.2771	
Re	<0L	<0L			<0L	<0L					0.0173	0.0576	
Rh	<0 L	<0L			<0L	<0L					0.0821	0.2738	
Ru	<0L	<0L			<0L	<0L				•	0.0350	0.1167	
Sb	<0L	<0L			<0L	<0L					0.0475	0.1584	
Se	≺ 0Ł	<0L			<dł< td=""><td><0L</td><td></td><td></td><td></td><td></td><td>0.1515</td><td>0.5050</td><td></td></dł<>	<0L					0.1515	0.5050	
Şi	832		11.7		1,461	1,908	26.6	*			0.0549	0.1830	
Şr	79	75	4.0		404	453	11.5				0.0004	0.0013	
Te	<0L	<0L			< 0∟	<0L					0.0950	0.3168	
Th	<0L	-70			<0L	< 0L					0.0734	0.2448	
Ti	<0L	<0L			(12)	(17)					0.0054	0.0179	
Ţl	<ol< td=""><td><0L</td><td></td><td></td><td><0L</td><td><dl< td=""><td></td><td></td><td></td><td></td><td>0.5492</td><td>1.8308</td><td></td></dl<></td></ol<>	<0L			<0L	<dl< td=""><td></td><td></td><td></td><td></td><td>0.5492</td><td>1.8308</td><td></td></dl<>					0.5492	1.8308	
Ü	(1,169)				11,547		13.6				0.5376	1.7920	
Ā	<0L	<0L			<ol< td=""><td><0L</td><td></td><td></td><td></td><td></td><td>0.0090</td><td>0.0301</td><td></td></ol<>	<0L					0.0090	0.0301	
Zn	343		20.6	•	323		16.2				0.0041	0.0136	
Zr	<0L	<0L				<0L					0.0076	0.0253	
P	11,370	11,580	1.8		20,473	20,315	0.8				0.6133	2.0442	
				_									

Note: 1) Values reliable to 2 1/2 significant digits. Bracketed results () are qualitative.

Data, including calibration/QC, archived File ICP-325-601- 01/05/93

lank is reported in ug/g "equivalence" to indicate blank effect on sample.

3) Sample results have not been adjusted for "blank" contribution.

4) At 50-100 times the D.L., precision is estimated at +/-10% and accuracy at +/-15%.

5) Sample DL (ug/g) = (DL in ug/mL) * (ug/g Factor)

6) Off-line IEC: Results within 50% DL potentially bias high.

7) 20% *** flag: RPD > 20% and both sample results > 3*QL.

Table 2-lg: SST Core 49, ICP Quarter Segment ID, Top and Bottom, Re-Homogenization Test, Fusion

ICP ANALYSIS REPORT -- KOH/Ni Fusions

PROJECT: SST

PROCEDURE: PNL-ALO-211

Analyzed Date: 12/02/92 M&TE: WA55672

Samp Log#: Dilution:	93- 9 01367-H1T 0 1.00	3- 11367-H2T 1.00		93- 01367-H18 1.00 2185.1 233	93- 01367-H2B 1.00		93- 01367-н3 1.00			
ug/g factor	2156.4	2151.4		2185.1	2193.9		2171.6			
ICP RUN #	232	21		ے کے	234		230		***Estima	steri***
	Sample	Dupl.	20	% Sample	Dupl.		20% Blank Flag ug/g	81k-Dup	DL	ar.
	ug/g	ug/g	RPD FL	ag ug/g	ug/g	RPD	20% Blank Flag ug/g	ug/g	ug/mL	ug/mL
Ag	<0L	<0L		<0L 53,141	<0L	****	<0L		0.0078	0.0261
AL	61,744	62,999	2.0	53,141	55,933	5.1	<0L		0.1829	0.6096
As	<0L	≪OL		<0L <0L (49) <0L 21,443	<0L		<0L		0.1014	0.3379
8	<0L	<0L		ΦL	<0L		<0L		0.0596	0.1987
8a	(54) <0L	(51)		(49)	(48)		<0L		0.0080	0.0267
Be	<0L	<0L		⊲υL	<0L		<0L		0.0032	0.0107
Ca	21,659	22,196	2.4	21,443	21,741	1.4	156		0.0016	0.0054
Cd	<ul< td=""><td><ul< td=""><td></td><td><111</td><td><d1< td=""><td></td><td><0L</td><td></td><td>0.0075</td><td>0.0250</td></d1<></td></ul<></td></ul<>	<ul< td=""><td></td><td><111</td><td><d1< td=""><td></td><td><0L</td><td></td><td>0.0075</td><td>0.0250</td></d1<></td></ul<>		<111	<d1< td=""><td></td><td><0L</td><td></td><td>0.0075</td><td>0.0250</td></d1<>		<0L		0.0075	0.0250
Ce	<0L	<0L <0L		<0L	<0L <0L		<0L		0.1019	0.3396
Ço	<ol< td=""><td><0L</td><td></td><td><0L</td><td><0L</td><td>_</td><td><0L</td><td></td><td>0.1865</td><td>0.6217</td></ol<>	<0L		<0L	<0L	_	<0L		0.1865	0.6217
Cr	255	263 154	3.1	241	248	3.1	<0L		0.0113	0.0378
Čn				(56)			<dl< td=""><td></td><td>0.0088</td><td>0.0294</td></dl<>		0.0088	0.0294
ДУ	<0L	<0L			<0L 12,811		< 0L		0.0053	0.0176
Fe		- 14,447	5.2			0.5	141		0.0100	0.0334
Ķ	NZA	N/A		H/A	N/A		N/A		0.3173	1.0576
La	<dl< td=""><td><0L</td><td></td><td><0L</td><td><0L</td><td></td><td><0€</td><td></td><td>0.0124</td><td>0.0414</td></dl<>	<0L		<0L	<0L		<0 €		0.0124	0.0414
Li	<0L	<0L		<0L	<0L		<0L		0.0074	0.0246
Mg	471		3.2	454	465	2.4	35		0.0003	0.0009
Mn	178	169	5.1	128	102	22.1			0.0010	0.0034
Мо	(32)	(41)		40L	<0L 89,172 <01		<0L		0.0128	0.0427
Na		90,543	0.3	90,231	89,172	1.2	744		0.0587	0.1957
Иd	.99	<0L					<dl< td=""><td></td><td>0.0496</td><td>0.1653</td></dl<>		0.0496	0.1653
Ni	N/A	N/A	17 /	N/A	N/A	•	N/A		0.0231	0.0768
Pb	625	745	17.0	646	650	0.6	<0L		0.0831	0.2771
Re	<0L	<0L		<0L	<0L		<0L		0.0173	0.0576
Rh	<0L	<0L		<0L	<0L		<0L		0.0821	0.2738
Ru	<0L	<0L		<0L	<dl< td=""><td></td><td>ФL</td><td></td><td>0.0350</td><td>0.1167</td></dl<>		ФL		0.0350	0.1167
Sb	<0L	<0L		<dl< td=""><td><0 L</td><td></td><td>≪DL</td><td></td><td>0.0475</td><td>0.1584</td></dl<>	<0 L		≪DL		0.0475	0.1584
Se	<0L	<0L		₹ 0£	<0L		< <u>0</u> L		0.1515	0.5050
Si	1,257	1,477		1,176	1,229		⊲DL		0.0549	0.1830
\$r	409	412	0.6	363	384	5.7	≪OL		0.0004	0.0013
Te	<0L	<0L		<0L	<0L		<0L		0.0950	0.3168
Th	<0L	<0L		<0L	<0L		<o∟< td=""><td></td><td>0.0734</td><td>0.2448</td></o∟<>		0.0734	0.2448
Ti Tl	<0L <0L	<0L		<0L	<0L		<0L		0.0054	0.0179
	12,251	<0L 13,080	6.5	<0L 10, 830	<0L 11,783	٠,	<0L		0.5492	1.8308
v	12,231 <0L	13,080 <0L	0.3			8.4	<0L		0.5376	1.7920
žn	317		16.0	<0L 278	<0L	1 7	<0L		0.0090	0.0301
	-61	-61			273	1.7	37		0.0041	0.0136
21	18 457	10 073	2 2	<0L 19,539	40L 18 47/	4.8	<0L		0.0076	0.0253
r	10,055	17,012	4.4	17,337	10,024	4.5	40 £		0.6133	2.0442

1) Values reliable to 2 1/2 significant digits. Bracketed results () are qualitative.
2) Blank is reported in ug/g "equivalence" to indicate blank effect on sample.
3) Sample results have not been adjusted for "blank" contribution.
4) At 50-100 times the D.L., precision is estimated at +/-10% and accuracy at +/-15%.
5) Sample DL (ug/g) = (DL in ug/mL) * (ug/g Factor)
6) Off-line IEC: Results within 50X DL potentially bias high.
7) 20% "*" flag: RPD > 20% and both sample results > 3*QL.

Data, including calibration/QC, archived File ICP-325-601- 12/02/92

Table 2-2a: SST Core 47, ICP Core Composite Acid Digestion

ICP ANALYSIS REPORT -- ACID DIGESTION ****** Sample Results ******

Project: SST

Procedure: PNL-ALO-211

Analyzed Oate: 01/18/93 M&TE: VA55672 Page 1 of 2

Sample S	Samp Log#: Dilution: ug/g factor	1.0 0 212.27	10.00 21 22.69		93-01358a2 1.00 217.58		4	93-01358 a 3 1.00 220.09				
Sample Sample Oupl. Ou	icy kun #	400	407		407			465				-
May May		C1 -			B 1						OL	ar.
Ag												
A1 74, 113 74, 745 0.9 71, 414 ol. 01. 0.1 1329 0.3079 A8 01. 01. 01. 01. 01. 01. 01. 01. 01. 01.		4 9/ 9	ng/g	2017	∪ g/ g	ug/g	72211	49/9	ug/g	720 1 f	ug/mL	ug/mL
A1 74, 113 74, 745 0.9 71, 414 ol. 01. 0.1 1329 0.3079 A8 01. 01. 01. 01. 01. 01. 01. 01. 01. 01.												******
As												
8 146 (161) 108 121 0.2596 0.1987 8a 56 59 5.4 58 (6) 0.0080 0.0267 8e 40L 40L 40L 0.0032 0.0107 Ca 19.503 21,991 12.8 20,541 317 0.0016 0.0075 Cd 13 (20) 111 40L 0.0075 0.0250 Ca 75 (273) 78 40L 0.1019 0.3396 Ca (59) 40L (50) 40L 0.1019 0.3396 Ca (59) 40L (50) 40L 0.1313 0.0378 Cu 65 (62) 54 40L 0.01133 0.0378 Cu 65 (62) 54 40L 0.01133 0.0373 Cu 65 (62) 15 40L 0.01373 1.0376 Fe 35,176 36,514 3.8 22,385				0.9	•							
Ba 56 59 5.4 58 (4) 0.0080 0.02267 Be 0L 0L 0L 0L 0L 0L 0.0032 0.0037 Ca 19,503 21,991 12.8 20,541 317 0.0016 0.0032 Cd 13 (200 11 0L 0.0075 0.0250 Ca 75 (273) 78 0L 0.1 0.1 0.1019 0.3396 Ca (59) 0L (500 0L 0.1 0.1 0.1 0.1 0.3396 Ca (59) 0L (500 0L 0.1 0.1 0.1 0.1 0.3396 Ca (59) 0L (500 0L 0.1 0.1 0.1 0.1 0.3 0.3 0.2 0.2 0.2 0.2 0.2 0.2 0.2 0.2 0.2 0.2												
Se				٠,								
Ca 19,503 21,991 12.8 20,541 317 0.0016 0.0025			-	5.4								
Cd 13 (20) 11								_				
Ce 75 (273) 78				12.8				_				0.0054
Co												0.0250
Cr 216 226 4.6 229								-				
Cu 65 (62) 54												0.6217
DY (2) (15) (2) (40 0.0053 0.0176 Fe 35,176 36,514 3.8 22,385 40 0.0100 0.0334 K 581 (954) 593 40L 0.3173 1.0576 La 81 93 14.6 81 40L 0.0124 0.0414 Li (4) (18) (4) 40L 0.0073 0.0009 Mn 162 167 3.0 106 (0) 0.0003 0.0009 Mn 162 167 3.0 106 (0) 0.0010 0.0034 Mn 43 (50) 43 40L 0.0128 0.0427 Ma 81,535 82,155 0.8 82,178 312 0.0587 0.1957 Md 130 (208) 129 40L 0.0496 0.1653 Ni 14,555 15,427 6.0 14,884 40L 0.0246 Pb 9,959 10,504 5.5 7,252 40L 0.0831 0.2771 Re (10) 40L (9) 40L 0.003 0.0576 Rh 40L 40L 40L 40L 40L 40L 40L 0.0032 0.1167 Sb 43 40L 47 40L 0.0320 0.1167 Sb 43 40L 47 40L 0.0320 0.1167 Sc 43 43 40L 47 40L 0.0320 0.1167 Sc 44 40L 40L 40L 40L 40L 40L 0.0559 0.1595 Si 1,659 1,701 2.6 2,148 99 0.0549 0.1830 Sr 188 190 1.2 190 1 0.0047 0.1535 Th 68 (234) 71 40L 0.00540 0.1830 Sr 188 190 1.2 190 1 0.00550 0.1167 Th 68 (234) 71 40L 0.00540 0.1830 Sr 188 190 1.2 190 1 0.0004 0.0013 Te 72 40L 71 40L 0.00540 0.1830 Sr 188 190 1.2 190 1 0.0004 0.0059 0.3168 Th 68 (234) 71 40L 0.00540 0.0059 U 10,454 11,443 11.4 10,955 40L 0.05376 1.7920 V 15 (21) 13 40L 0.0076 0.0253 P 18,434 19,039 3.3 18,447 40L 0.0064 0.0073 2.0442				4.6								0.0378
Fe 35,176 36,514 3.8 22,385 40 0.0100 0.0334 K 581 (954) 593 40L 0.3173 1.0576 La 81 93 14.6 81 40L 0.0124 0.0414 Li (4) (18) (4) 40L 0.0 0.0074 0.0246 Mg 444 481 8.3 493 68 0.0003 0.0009 Mn 162 167 3.0 106 (0) 0.0011 0.0034 Mo 43 (50) 43 40L 0.0128 0.0427 Ma 81,535 82,155 0.8 82,178 312 0.0587 0.1957 Md 130 (208) 129 40L 0.0587 0.1957 Md 130 (208) 129 40L 0.0496 0.1653 Ni 14,555 15,427 6.0 14,884 40L 0.0231 0.0768 Pb 9,959 10,504 5.5 7,252 40L 0.0831 0.2771 Re (10) 40L 40L 40L 40L 0.0831 0.2771 Re (10) 40L 40L 40L 40L 0.0350 0.1167 Sb 43 40L 40L 40L 40L 40L 0.0350 0.1167 Sb 43 40L 40L 40L 40L 40L 0.0350 0.1167 Sb 43 40L 47 40L 0.0475 0.1584 Se 40L 40L 47 40L 0.0475 0.1584 Se 40L 40L 47 40L 0.0475 0.1584 Se 40L 40L 47 40L 0.0475 0.1584 Se 40L 40L 47 40L 40L 40L 0.0549 0.1630 Th 68 (234) 71 40L 40L 40L 40.00137 0.2739 Tl 40L												0.0294
X 581 (954) 593 □ 0.3173 1.0576 La 81 93 14.6 81 □ 0.0124 0.0244 Li (4) (18) (4) □ □ 0.0246 0.0246 Mg 444 481 8.3 493 68 0.0003 0.0009 Mn 162 167 3.0 106 (0) 9.001 0.0034 Mn 43 (50) 43 □ □ 0.0231 0.0427 Ma 81,535 32,155 0.3 82,178 312 0.0587 0.1957 Md 130 (208) 129 □ □ 0.0496 0.1653 Ni 14,555 15,5427 6.0 14,884 □ □ 0.0231 0.2771 Re (10) □ (9) □ □ 0.0831 0.2771 Re (10) □ (9) □ □ 0.0351 0.2773 Ru □ □ □ □ □ <td></td> <td>(2)</td> <td></td>		(2)										
La 81 93 14.6 81		35,176		3.8				_				0.0334
Li (4) (18) (4) (4)								<0L				
Mg 424 481 8.3 493 68 0.0003 0.0009 Mn 162 167 3.0 106 (0) 0.0010 0.0010 0.0010 0.0021 0.0021 0.0021 0.0021 0.0021 0.0021 0.0023 0.0027 0.0023 0.1957 0.1957 0.1957 0.1957 0.00231 0.0587 0.1957 0.1957 0.00231 0.0587 0.1957 0.00231 0.0587 0.1957 0.00231 0.0587 0.1957 0.00231 0.0587 0.1957 0.00231 0.0587 0.1957 0.00231 0.0587 0.1957 0.00231 0.0768 0.00231 0.0768 0.00231 0.0771 0.0768 0.00231 0.2771 0.0771 0.0 0.00331 0.2771 0.0 0.00331 0.2771 0.0 0.00331 0.2771 0.0 0.00331 0.2771 0.0 0.00331 0.2771 0.0 0.00330 0.1167 0.0 0.03330 0.1167 0.0 0.03330 0.1167 0.0 0.03330 0.1167 0.0 0.00330 0.1515 0.0549 <				14.6				<0L				0.0414
Mn 162 167 3.0 106 (0) 0.0010 0.0034 Mo 43 (50) 43 (0L 0.0128 0.0427 Ma 81,535 82,155 0.8 82,178 312 0.0587 0.1957 Md 130 (208) 129 (0L 0.0466 0.1653 Mi 14,555 15,427 6.0 14,884 (0L 0.0231 0.0768 Pb 9,959 10,504 5.5 7,252 (0L 0.0831 0.2771 Re (10) (0L (9) (0L 0.0173 0.0576 Rh (10) (0L (0L (0L 0.0821 0.2738 Ru (0L (0L 0.0821 0.2738 Ru (0L (0L (0L (0L 0.0350 0.1167 5.5 5.5 43 43 (0L (0L (0L (0L (0L (0L (0L (0L (0L (0L												
Mo								68				
Na 81,535 82,155 0.8 82,178 312 0.0587 0.1957 Nd 130 (208) 129				3.0				(0)				
Nd												0.0427
Ni 14,555 15,427 6.0 14,884				0.8				312				
Pb 9,959 10,504 5.5 7,252 40L 0.0831 0.2771 Re (10) 40L (9) 40L 0.0173 0.0576 Rh 40L 40L 40L 40L 0.0821 0.2738 Ru 40L 40L 40L 40L 0.0350 0.1167 Sb 43 40L 47 40L 0.0475 0.1584 Se 40L 40L 40L 40L 0.1515 0.5050 Si 1,659 1,701 2.6 2,148 99 0.0549 0.1830 Sr 188 190 1.2 190 1 0.004 0.0013 Te 72 40L 71 40L 0.0950 0.3168 Th 68 (234) 71 40L 0.0734 0.2448 Ti 55 63 14.4 64 40L 0.0074 0.0179 Tl 40L 40L								۰ <0L				
Pb 9,959 10,504 5.5 7,252 40L 0.0831 0.2771 Re (10) <0L	Ni							<0L				0.0768
Re (10) <0L	Pb			5.5				٩				0.2771
Rh		(10)	≪0 L		(9)			<0L			0.0173	0.0576
8u <0L	Rh	≺0L	40 ار		<0ኒ			<0L	•		0.0821	0.2738
Sb 43 OL 47 OL 0.0475 0.1584 Se OL OL OL OL 0.1515 0.5050 Si 1,659 1,701 2.6 2,148 .99 0.0549 0.1830 Sr 188 190 1.2 190 1 0.004 0.0013 Te 72 OL 71 OL 0.0950 0.3168 Th 68 (234) 71 OL 0.0734 0.2448 Ii 55 63 14.4 64 OL OL 0.0734 0.2448 Ii 55 63 14.4 64 OL OL 0.0734 0.2448 Ii 55 63 14.4 64 OL OL 0.05492 1.8308 U 10,454 11,643 11.4 10,955 OL 0.5376 1.7920 V 15 (21) 13 OL 0.0090 0.0301	Ru	· <0L	< 0L		<0L			40 £			0.0350	
Se <0L <0L <0L <0L <0L <0L <0L <0.550 \$i 1,659 1,701 2.6 2,148 .99 0.0549 0.1830 \$r 188 190 1.2 190 1 0.004 0.0013 \$r 72 <0L	Sb	43	<0L		47			≪OL			0.0475	
\$i 1,659 1,701 2.6 2,148 99 0.0549 0.1830	Se	ΦL	<0L		<0L			√0 Ł				
Sr 188 190 1.2 190 1 0.0004 0.0013 Te 72 <0L	Sí	1,659	1,701	2.6	2,148			. 99			. 0.0549	
Te 77	Sr	188	190		190	•						
Th 68 (234) 71	Τœ	72	<0L		71			<0L				
T1 55 63 14.4 64 <0L	Th	68	(234)		71			<0L				
Tt	Ti	55	63	14.4	64			• -				
U 10,454 11,643 11.4 10,955				-								1.8308
V 15 (21) 13 <0L				11.4								1.7920
Zn 267 398 48.9 246 3 0.0041 0.0136 Zr (4) <ol (3)="" 0.0076="" 0.0253<br="" <ol="">P 18,434 19,039 3.3 18,447 <ol 0.6133="" 2.0442<="" td=""><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td>• -</td><td></td><td></td><td></td><td></td>								• -				
2r (4) <0L (3) <0L 0.0076 0.0253 P 18,434 19,039 3.3 18,447 <0L 0.6133 2.0442	Žn			48.9								
P 18,434 19,039 3.3 18,447 <0L 0.6133 2,0442								_				
				3.3								
			,		,						410.55	

Oats, including calibration/QC, archived File ICP-325-601 01/18/93

Note: 1) Values reliable to 2 1/2 significant digits. Bracketed results () are qualitative.
2) Blank is reported in ug/g "equivalence" to indicate blank effect on sample.
3) The process "Blank" has not been subtracted from the "Sample & Ouplicate" results.
4) At 50-100 times the D.L., pracision is estimated at +/-10% and accuracy at +/-15%.
5) "Estimated" Sample Detection Limit (ug/g) = (DL in ug/mt) * (ug/g factor)
6) Off-line IEC: Results within 50% DL potentially bias high.

Table 2-2b: SST Core 47, ICP Core Composite Acid Digestion QC Results

Project: SST Procedure: PNL-ALG-211 Analyzed: 01/18/93 M&TE: WAS5672 Page 2 of 2

Samp Log#:	93-01358a4	93-01358a5
Dilution:	1.00	1.00
ug/g Factor:	231.27	100.46
ICP Run #:	470	466
	<	

[CP Run #:			7 #:	470					466						
	Ave al&a2 ug/g	RPD	20% Flg	Spike Added ug/g	Spike+ Sample ug/g	Spike+ Sample ug/g	20if	%Rec	Spk Flag	Spike STD ug/mL	Spike Control ug/mL	Spike Control ug/mL	%Dif	#Rec	
Ag Al	N/A 72,863	3.4		115.1 575.5	82 85,338			72 N/A	\ #	50.0 250.0	41.7 231.1			83.4 92.4	
As	N/A 127	29.6		460.4	390 <i>7</i> 5			85		200.0	194.5			97.3	
8 8a	57	3.3		115.1	176			103		50.0	56.1			112.1	
8e	N/A			11.5	12			102		5.0	5.1			102.3	
Ca	20,022	5.2		1,151.0	21,476			N/A	#	500.0	361.8			72.4	
Cd Ce Co	12 77 N/A	17.5 3.5		57.6	64 86 (70)			91		25.0	25.1			100.3	
Cr Cu	223 59	5.8 18.3		115.1	334 61			97		50.0	49.9			99.8	
ОУ	N/A				(3)										
Fe	28,781	44.4	*	115.1	20,165			N/A	#	50.0	22.3			44.6	
K La	587 81	2.1 0.8		2,302.0	2,854 79			98		1000.0	1031.8			103.2	
Ļi	N/A	٠.٠			(Ś)										
Hg	469	10.4			583 ´										
Mn	134	42.2	*	23.0	125			-38	#	10.0	10.1			101.3	
Мо	43	1.1			47										
Na Nd	81,856 129	0.8		2,302.0	86,508			A/K	#	1000.0	853.1			85.3	
Ni	14,720	0.3 2.2		115.1	132 14, <i>7</i> 21			N/A	4	50.0	50.8			101.6	
Pb	8,606	31.5	*	1,151.0	8,195			-36		500.0	493.2			98.6	
Яe	N/A			.,	(10)				"	300.0	4,4.4			70.0	
Rh	N/A				<0L										
Ru	A/K				<0r										
Sb Se	45 H/A	10.6		1 151 0	54					500.0	777 0				
Si	1,903	25.7	*	1,151.0 1,151.0	694 2,236			60 29		500.0 500.0	332.9 739.3			66.6	
Sr	189	1.0		1,151.0	192			47		300.0	737.3			147.9	
Te	72	2.2			85										
Τħ	69	4.3			86										
Ti	59	15.1			145										
Tl U	N/A 10,704	4.7		2,302.0	<0L			0.4	м	4000 0				·	
٧	10,704	14.9		115.1	12,677 107			86 81	#	1000.0 50.0	976.2 51.3			97.6	
Žn	256	8.5		11344	221			G.I		טייטנ	2.13			102.6	
Zr	N/A			115.1	10			8		50.0	47.1			94.1	
P	18,441	0.1		4 454 6	19,299										
8 i	A/K			1,151.0						500.0					

Note: 1) Values reliable to 2 1/2 significant digits. Bracketed results () are qualitative.

²⁾ At 50-100 times the 0.L., precision is estimated at +/-10% and accuracy at +/-15%.

3) The process "Blank" has been subtracted from the "Spike Control" results.

4) Spike Flag (#): Spike is less than 25% of sample concentration; %recovery for information only.

5) 20% Flag (*): RPO > 20% and both sample and duplicate results > 3*qL.

6) If spike is <10% of sample concentration, %Rec is not calculated as indicated by the "n/a".

Table 2-2c: SST Core 48, ICP Core Composite Acid Digestion

ICP ANALYSIS REPORT -- ACID DIGESTION Sample Results

Project: SST Procedure: PNL-ALO-211 Analyzed Date: 01/18/93 M&TE: WA55672

Page 1 of 2

Samp Log#:	93-01363a1	93-01363#2	93-01363a2
Dilution:	1.00	1.00	10.00
ug/g factor	202.72	217.25	2172.50
ICP Run #	476	475	474

ICP Run #	476			475	474					***Estim	ated***
		1 .								OL	QL
	Sample	Sample	20 if	Dupl.	Oupl.	wa i i	Blank	8lank	No. 2 a		
	ug/g	ug/g	AU 11	ug/g	ug/g	2Dif	ug/g	ug/g	2Dif	ug/mL	ug/mL
Ag	<0L			∢٥L	≪OL					0.0078	0.0261
ΑĨ	6,241			6,597	6,512	1.3				0.1829	0.6096
As	Ć∢0L			≺0L	≪ 0L					0.1014	0.3379
8	109			161	(157)					0.0596	0.1987
Ba	48			54	(55)					0.0080	0.0267
₿e	<0L			∢٥L	<0L					0.0032	0.0107
Ca	14,376			10,734	11,407	6.3				0.0016	0.0054
Çd	7			10	<0L					0.0075	0.0250
Ce	<0∟			(34)	<0L					0.1019	0.3396
Ça	<0L			<0L	<0L					0.1865	0.6217
Сr	212			208	210	1.2				0.0113	0.0378
Çu	14			28	(23)					0.0088	0.0294
ολ	(1)			(3)	(16)					0.0053	0.0176
۶e	13,929			26,461	26,521					0.0100	0.0334
ĸ	609			665	(1,046)					0.3173	1.0576
La	(6)			(7)	<0L					0.0124	0.0414
Li	9			9	(22)					0.0074	0.0246
Мg	527			497	521	4.8				0.0003	0.0009
Mri Mo	54 31			138 31	138 (33)	0.4				0.0010	0.0034
Na Na				93,517		1.0				0.0128	0.0427
ЬК	81,622 43			45	92,587 (130)					0.0587	0.1957
Ni	16,307			14.732	15,073	2.3				0.0496 0.0231	0.1653 0.0768
Pb	586			626	627	0.1				0.0831	0.2771
Re	(6)			(6)	<0L	4.1	-			0.0173	0.0576
Rh.	<òt. ⊂			<ÔĽ	∢0L					0.0821	0.2738
Ru	<0L			(11)	≪OL					0.0350	0.1167
Sb	46			67	<0L					0.0475	0.1584
Se	≺OĹ			<0L	<ol< td=""><td></td><td></td><td></td><td></td><td>0.1515</td><td>0.5050</td></ol<>					0.1515	0.5050
Si	1,229			1,394	. 1,174	15.8				0.0549	0.1830
Sr	397			492	486	1.3				0.0004	0.0013
Τ e	<0L			<0L	<0L					0.0950	0.3168
Th	(30)			(49)	(229)					0.0734	0.2448
Ti	8			13	<ql< td=""><td></td><td></td><td></td><td></td><td>0.0054</td><td>0.0179</td></ql<>					0.0054	0.0179
Ťt	≪OL			<0L	<0L					0.5492	1.8308
u	12,684			17,377	18,147	4.4				0.5376	1.7920
V	(4)			9	<ol< td=""><td></td><td></td><td></td><td></td><td>0.0090</td><td>0.0301</td></ol<>					0.0090	0.0301
2n	196			195	277	41.7				0.0041	0.0136
Zr P	(5)			10 501	40L					0.0076	0.0253
g Si	14,487			19,591	19,591	0.0				0.6133	2.0442
01											

Note: 1) Values reliable to 2 1/2 significant digits. Bracketed results () are qualitative.
2) Blank is reported in ug/g "equivalence" to indicate blank effect on sample.
3) The process "Blank" has not been subtracted from the "Sample & Duplicate" results.
4) At 50-100 times the 0.L., precision is estimated at +/-10% and accuracy at +/-15%.
5) "Estimated" Sample Detection Limit (ug/g) = (DL in ug/mL) * (ug/g Factor)

- 6) Off-line IEC: Results within 50% OL potentially bias high.

Data, including calibration/OC, archived File ICP-325-601 01/18/93

Table 2-2d: SST Core 48, ICP Core Composite Acid Digestion QC Results

ICP ANALYSIS REPORT -- ACID DIGESTION

Project: Procedure: SST PNL-ALO-211 Analyzed: 01/18/93 M&TE: WA55672 Page 2 of 2

Samp Log#: Dilution: ug/g Factor: [CP Run #:				1<	93-01363a4 1.00 233.05 477				. 1					. 1
	Ave al&a2 ug/g	RPD	20% Flg	Spike Added ug/g	Spike+ Sample ug/g	Spike+ Sample Ug/g	20 if	%Rec	Spk Flag	Spike STD Ug/mL	Spike Control ug/mL	Spike Control ug/mL	XD if	Жес
Ag Al As B	N/A 6,419 N/A 135	5.6 38.5		116.0 579.9 463.9	84 12,226 399 114			72 H/A 86	#	50.0 250.0 200.0				
Ba Be Ca	51 N/A 12,555	12.5 29.0	*	116.0 11.6 1,159.9	167 10 13,305			100 87 N/A	#	50.0 5.0 500.0				
Cd Ce Co	8 N/A N/A 210	2.3		58.0 116.0	58 (30) <0L 290			86 69		25.0 50.0				
Cu Oy Fe K	21 N/A 20,195 637	67.3 62.1 8.7	•	116.0 2,319.7	21 (2) 14,179 2,714			N/A 90	*	50.0 1000.0				
La Li Mg Mn	N/A 9 512 96	1.9 5.9 87.8	•	23.2	(7) 7 503 74			-94	#	10.0				
Mo Na Nd Ni	31 87,570 44 15,520	0.1 13.6 5.3 10.1		2,319.7 116.0	33 77,008 46 14,743			N/A N/A		1000.0 50.0				्र
Pb Re Rh Ru	606 N/A N/A N/A	6.7		1,159.9	1,591 (7) <ol <ol< td=""><td></td><td></td><td>85</td><td></td><td>500.0</td><td></td><td></td><td></td><td></td></ol<></ol 			85		500.0				
Sb Se Si Sr Te Th	56 N/A 1,312 444 N/A N/A	37.1 12.5 21.5	•	1,159.9 1,159.9	58 696 2,472 454 <0L (52) 8			60 100		500.0 500.0				
T l U V Zn	N/A 15,030 N/A 196	31.2 0.2	*	2,319.7 116.0	<0L 16,945 99 197			83 85	#	1000.0 50.0				
Žr P Bi	N/A 17,039 N/A	29.9	*	116.0 1,159.9	99 13,328			86		50.0 ×				

Note: 1) Values reliable to 2 1/2 significant digits. Bracketed results () are qualitative.
2) At 50-100 times the 0.1., precision is estimated at +/-10% and accuracy at +/-15%.
3) Spike Flag (#): Spike is less than 25% of sample concentration; %recovery for information only.
4) 20% Flag (*): RPD > 20% and both sample and duplicate results > 3*QL.
5) If spike is <10% of sample concentration, %Rec is not calculated as indicated by the "n/a".

Table 2-2e: SST Core 49, ICP Core Composite Acid Digestion

ICP ANALYSIS REPORT -- ACID DIGESTION Sample Results

Project: SST Procedure: PNL-ALO-211 Analyzed Date: 01/18/93 M&TE: WA55672

Page 1 of 2

Ug/g Factor 200.88 198.93 200.07 CP Run # 457 458 455 ****Est** DL	***Estimated***				
Sample Sample Dupt. Oupt. Stank Stank ug/g ug/g 20if ug/g ug/g 20if ug/g ug/g 20if ug/mL	QL ug/mL				

41 00 000	0.0261				
0.102	0.6096				
4.1015	0.3379				
4- 4- 4- 4- 4- 4- 4- 4- 4- 4- 4- 4- 4- 4	0.1987				
	0.0267				
0.003	0.0107				
4.000	0.0054				
Cd 9 7 40L 0.0075 Ce (46) (26) 40L 0.1015	0.0250 0.3396				
Co (71) (37) <0L 0.1865					
Cr 202 162 <0L 0.0113	0.6217 0.0378				
Cu 25 24 <0L 0.0088	0.0294				
9y <u>40L</u> 40L 40L 0.0053	0.0176				
Fe 8,393 5,899 17 0.0100	0.0334				
K 460 354 <al 1173<="" do="" n="" td=""><td>1.0576</td></al>	1.0576				
La 51 38 «OL 0-01726	0.0414				
Li (3) (2) «oL 0.0072	0.0246				
Mg 325 267 58 0.0003	0.0009				
Mn 57 39 «OL 0.0010	0.0034				
Mo 45 35 45 0_0128 	0.0427				
Na 65,806 96,785 168 0.0587	0.1957				
Nd 90 68 <ol #="" 0.0498<="" td=""><td>0.1653</td>	0.1653				
Ni 13,115 10,620 <0. 0.0231	0.0768				
Pb 999 728 <0L 0.0831	0.2771				
Re (8) (5) <0L 0.0173	0.0576				
Rh <0L	0.2738				
Ru <0L <0L <0L <0L 0.0350	0.1167				
Sb (29) (26) ≪0L 0.0475	0.1584				
\$	0.5050				
\$1 1,515 1,272 84 0.0549	0.1830				
\$r 167 142 1 0.0004	0.0013				
Te 93 69 <0L 0.0950	0.3168				
Th (37) (16) <0L 0.0734	0.2448				
71 7 4 <0L 0.0054	0.0179				
0.3476	1.8308				
U 7,102 5,429 <0L 0.5376	1.7920				
77. 0.0040	0.0301				
0.0041	0.0136				
0.0070	0.0253				
P 11,710 27,108 <0L 0.4133	2.0442				

Oata, including calibration/OC, archived File ICP-325-601 01/18/93

Note: 1; Values reliable to 2 1/2 significant digits. Bracketed results () are qualitative.
2) Blank is reported in ug/g "equivalence" to indicate blank effect on sample.
3) The process "Blank" has not been subtracted from the "Sample & Duplicate" results.
4) At 50-100 times the 0.L., precision is estimated at +/-10% and accuracy at +/-15%.
5) "Estimated" Sample Detection Limit (ug/g) = (DL in ug/mL) * (ug/g Factor)
6) Off-line IEC: Results within 50% DL potentially bias high.

Table 2-2f: SST Core 49, ICP Core Composite Acid Digestion QC Results

ICP ANALYSIS REPORT -- ACID DIGESTION ****** QC Results *******

Project: SST Procedure: PNL-ALO-211

Analyzed: 01/18/93 M&TE: 4455672 Page 2 of 2

Samo Log#: Oilution: Ug/g Factor: [CP Run #:				l <i>c</i>	93-01371a4 1.00 200.40 459				73-01371a5 1.00 100.46 456				
	Ave al&a2 ug/g	RPD	20% Flg	Spike Added ug/g	Spike+ Sample ug/g	Spike+ Sample ug/g	220 if	Sok Zec Flag	Spike STO ug/mL	Spike Control ug/mL	Spike Control ug/mL	2Dif	#Rec
Ag Al As B	N/A 83,696 N/A 55	29.1 34.0	•	99.7 498.7 399.0	71 105,460 319 79			71 N/A # 80	50.0 250.0 200.0	40.1 228.6 184.5	*****	****	80.3 91.5 92.3
Ba Be Ca Cd	28 N/A 12,336 8	25.8 24.5 31.7	•	99.7 10.0 997.4 49.9	140 10 15,239 54			112 103 N/A # 92	50.0 5.0 500.0 25.0	54.7 4.8 380.7 23.3			109.4 96.5 76.1 93.4
Co	N/A N/A 182 24	22.1 6.1	=	99.7	(45) (84) 302 34			121	50.0	46.9			93.7
Dy Fe K La	H/A 7,146 407 44 H/A	34.9 26.0 29.6	*	9 9. 7 1,9 94.8	<0L 7,790 2,440 46 (4)			N/A # 102	50.0 1000.0	40.1 1003.2			80.2 100.3
PK OK OK	296 48 40 81,295	19.8 36.3 26.4 38.1	* *	19.9 1.994.8	353 71 49 75,208			118 ×/a #	10.0	9. <i>7</i> 1034.3			97.5
Nd Ni Pb Re	79 11,867 864 H/A	28.0 21.0 31.4	*	99.7 997.4	89 13,631 1,805 (9)			N/A # 94	50.0 500.0	48.8 464.1			97.5 92.8
Rh Ru Sb Se	A\K A\H A\H A\H			997.4	<0L <0L 41 579			58	500.0	320.4			64.1
Si Sc Te Th Ti	1,393 154 81 H/A 5	17.5 16.7 29.3		997.4	2,168 183 107 (41)			78	500.0	588.1	•		117.6
ti u v Zn	H/A 6,266 H/A 278	26.7	•	1,994.8 99.7	6 ≺0L 9,113 92 317			143 92	1000.0 50.0	964.5 48.8			96.4 97.6
Zr P Bi	N/A 19,409 H/A	79.3	•	99.7 997.4	14 14,204			14	50.0 500.0	49.0			98.1

Note: 1) Values retiable to 2 1/2 significant digits. Bracketed results () are qualitative.

2) At 50-100 times the D.L., precision is estimated at +/-10% and accuracy at +/-15%.

3) The process "Blank" has been subtracted from the "Spike Control" results.

4) Spike Flag (#): Spike is less than 25% of sample concentration; %recovery for information only.

5) 20% Flag (*): RPD > 20% and both sample and duplicate results > 3*QL.

6) If spike is <10% of sample concentration, %Rec is not calculated as indicated by the "n/a".

Table 2-2g: SST C-109, ICP Liquid Composite Acid Digestion

1CP ANALYSIS REPORT -- ACID DIGESTION Sample Results

Project: SST Procedure: PNL-ALO-211 Analyzed Date: 01/22/93 M&TE: WA55672 Page 1 of 2

Samo Log#: Oilution: ug/g Factor [CP Run #	93-01354a1 1.00 169.72 507		•	93-01354a2 1.00 164.31 508		•	93-01354 a3 1.00 167.24 505			TTTG-si-	
							,4,			TTESTIM OL	QL.
	sampi e	Sample		Oupl.	Օսթ:		Blank	81 ank			
	ug/g	ug/g	720 i f	ug/g	ug/g	≃D if	ug/g	ug/g	2Dif	ug/mt	ug/mL
λg	<0L		•••	٠٠٠			«OL				
ÂĹ	161			153			۵۲ ۱			0.0081	0.0272
As	<0L			۰۵L			<0L			0.1904 0.1055	0.4345 0.3517
9	93			40			۵۲.			0.0620	0.2068
8a	(4)			(3)			∢OL			0.0083	0.0278
8e	<01			۱٥٠ ×			∢0L			0.0033	0.0276
Ça	213			205			234			0.0033	0.0056
Çd	<0L			<ql< td=""><td></td><td></td><td>«OL</td><td></td><td></td><td>0.0078</td><td>0.0260</td></ql<>			«OL			0.0078	0.0260
Ce	<0L			<ql< td=""><td></td><td></td><td>٠٥L</td><td></td><td></td><td>0.1060</td><td>0.3534</td></ql<>			٠٥L			0.1060	0.3534
Co				<0L			∢OL			0.1941	0.6471
Cr	289			293			(2)			0.0118	0.0393
Cu	< 0 L			<0L			<0L			0.0092	0.0306
Oy	₹ 0६			40 L			< 0L			0.0055	0.0183
f∈				1,698			≪OL			0.0104	0.0347
K	835			844			<0L			0.3302	1.1008
Lạ	«OL			< 0∟			< 0L			0.0129	0.0430
Ļi	<0L			<0L			<0L			0.0077	0.0256
Ма	26			26			1			0.0003	0.0009
Жn				(0)			<ol< td=""><td></td><td></td><td>0.0011</td><td>0.0035</td></ol<>			0.0011	0.0035
Жа				40			40 ٤			0.0133	0.0445
На	96,947			96,936			<0 L			0.0611	0.2037
Mq				≪ 0 L			< 0 ل			0.0516	0.1720
Ni.	340			347			<0ኒ			0.0240	0.0800
Pb	≪0L			<0L			<0L			0.0865	0.2884
Я e				<0L			<0L			0.0180	0.0599
Rh				<0L			<0L			0.0855	0.2850
uR d2				<0L			<0L			0.0364	0.1235
26				<0L			٠QL			0.0495	0.1649
ser Si	74			<0L			<0L			0.1577	0.5256
, 31		٠.		63			«OL			0.0571	0.1904
Te				1 <0L			«OL			0.0004	0.0013
Th.	(0 L						<0L			0.0989	0.3297
Ti	₹ 0 L			<0L <0L			≪OL			0.0764	0.2548
Ti	<0L						<0L			0.0056	0.0187
ů`	√0L			<0ί. <0ί.			≪OL			0.5717	1.9056
Ÿ	√ 0L			-αι -αι			<0L			0.5595	1.8651
Zn				11			٠٥L			0.0094	0.0313
2.7	-{OL			<0L			«OL			0.0043	0.0142
P	4, 166			4,229			≺0L			0.0079	0.0263
r (4,447			40 ٤			0.6383	2.1277

Note: 1) Values reliable to 2 1/2 significant digits. Bracketed results () are qualitative.

2) Blank is reported in ug/g "equivalence" to indicate blank effect on sample.

3) The process "flank" has not been subtracted from the "Sample & Duplicate" results.

4) At 50-100 times the 0.L., precision is estimated at */-10% and accuracy at */-15%.

5) "Estimated" Sample Detection Limit (ug/g) = (DL in ug/mL) * (ug/g factor)

6) Off-line IEC: Results within 50% DL potentially bias high.

Oata, including calibration/GC, archived file ICP-325-601 01/22/93

01/26/93

Table 2-2h: SST C-109, ICP Liquid Composite Acid Digestion QC Results

ICP ANALYSIS REPORT -- ACID DIGESTION

07 04751-7

Project: SST

Procedure: PNL-ALO-211

3.5

Zn Zr P Analyzed: 01/22/93 M&TE: WA55672 Page 2 of 2

07 0175/-6

	ug/	amp Log# Dilution g factor CP Run #	: :	93-01354a4 1.00 167.53 509				1	9	73-01354a5 1.00 100.00 506				
	Ave a1&a2 ug/g	201 RPD Ft		Spike+ Sample ug/g	Spike+ Sample ug/g	20 if	ХЯес	Spk Flag	Spike STD ug/mL	Spike Control ug/mL	Spike Control ug/mL	%Dif	%Rec	
Ag Al As	N/A 157 N/A	4.9	83.5 417.7 334.2	86 675 318 49			103 124 95		50.0 250.0 200.0	43.3 244.5 202.5			86.5 97.8 101.2	
8 Ba Be Ca Cd	67 N/A N/A 209 N/A	4.0	83.5 8.4 835.4 41.8	125 11 1,326 53			149 128 134 126		50.0 5.0 500.0 25.0	49.0 5.0 500.5 24.4			98.0 99.5 100.1 97.6	
Ce Cu Cu	N/A N/A 291 N/A	1.2	83.5	<0L <0L 413 (5)			146		50.0	53.6			107.1	
Oy Fe K La Li	N/A 1,675 840 N/A N/A	2.8 1.0	83.5 1,670.8	<0L 2,087 3,042 <0L <0L			N/A 132	#	50.0 1000.0	64.3 1024.8			128.5 102.5	
Mg Mn Mo	26 N/A 40	0.8 1.1	16.7	51 22 43			130		10.0	10.4			104.2	
Na Nd	96,942 N/A	0.0	1,670.8	100,168 <0L			N/A	#	1000.0	1007.9			100.8	1
Xí Pb Re Rh	344 N/A N/A N/A N/A	1.9	83.5 835.4	644 1,011 <0L <0L <0L			360 121	# .	50.0 500.0	49.5 487.1			99.1 97.4	
Sb Se Si Sr Te Th	A/A A/A B3 1 A/A A/A	16.6 70.4	835.4 835.4	 <0L 522 1,721 3 <0L <0L 			62 198		500.0 500.0	345.4 764.5			69.1 152.9	
Ti Tl U	H/A H/A H/A		1,670.8	(1) <0L 1,932			116		1000.0	920.3			92.0	

124

117

50.0

50.0

500.0

50.4

44.0

100.8

87.9

14

98

4,356

83.5

835.4

Note:

¹⁾ Values reliable to 2 1/2 significant digits. Bracketed results () are qualitative.
2) At 50-100 times the D.L., precision is estimated at +/-10% and accuracy at +/-15%.
3) The process "Blank" has been subtracted from the "Spike Control" results.
4) Spike Flag (#): Spike is less than 25% of sample concentration; %recovery for information only.
5) 20% Flag (*): RPO > 20% and both sample and duplicate results > 3*qL.
6) If spike is <10% of sample concentration, %Rec is not calculated as indicated by the "n/a".

Table 2-2i: SST Core 48, ICP Quarter Segment 1D, Top, Homogenization Test, Acid Digestion

ICP ANALYSIS REPORT -- Acid Digestion

PROJECT: SST Analyzed Date: 11/13/92 M&TE: VAS5672 PROCEDURE: PNL-ALO-211

	93-	93-		93 -	93-		93 -	93 -					
Samp Log#:	1361+A1T	1361-A1T		1361-A2T	1361-A2T		1361-A3	1361-A3					
Dilution:	1,00	5.00		1.00	5.00		1.00	5.00					
ug/g factor		997.4		199.9	999.5		199.7	998.5					
(CP Run #	152	170		153	171		151	169	***Estin	ated***	1361-A1T		
									OL	QL	1361-A2T		
	Sample	Sampie		Sample	Sampie		Blank	8 lank			Average		20%
	ug/g	ug∕g	20if	ug/g	ug/g	20 if	ug/g	ug/g	ug/mL	ug/mL	ug/g	RPO	flg
			*					*******			•		•••
Ag	<ol< td=""><td><0L</td><td></td><td>. ≪OL</td><td>- «OL</td><td></td><td><0L</td><td>∢۵۲</td><td>0.0078</td><td>0.0261</td><td>A/K</td><td></td><td></td></ol<>	<0L		. ≪OL	- «OL		<0L	∢۵۲	0.0078	0.0261	A/K		
AL	8,725	9,042	3.4	7,891	7,866	0.3	<0L	≪OL	0.1829	0.6096	8,308	10.0	
As	≪UL	<0L		. ∢OL	<0L		٩L	<0L	0.1014	0.3379	H/A		
9	111	(114)	• •	81	(82)		66	(62)	0.0596	0.1987	96	30.8	
8a	45	46	2.4	43	42	1.7	<0L	<0L	0.0080	0.0267	44	6.2	
Be □	<0L	40L					<0L	<0L	0.0032	0.0107	A/A	_	
Ca	15,260 8	17,072	11.9	14, 193	15,230	7.3	496	727	0.0016	0.0054	14,725	7.2	
Cd Ce	જાર	(8) <0L		- 6	≪0L ≪0L		<0L	٩L	0.0075	0.0250		34.3	
· Co	√0L	40 L		<0ኒ <0L			≪L	<0L	0.1019	0.3396	N/A		
Cr	218	227	4.3	194	<0L 196	0.9	ØL ØL	<0L	0.1865 0.0113	0.6217	N/A		
Cri	14	(10)	7.3	12	≪0L	U. 7	- ⟨0 Γ - ⟨0 Γ	<0Γ	0.0088	0.0294	206	11.5	
0y	√ 0L	<0L		۷0L	<0L		اراد ا	<0£	0.0053	0.0274	13	22.3	
Fe	23,845	24,893	4.4	13.747	13,727	0.1	11	₹0 L	0.0033	0.0334	N/A 18,796	53.7	
ĸ	648	(571)	7,7	527	(393)		άr,	√0 L	0.3173	1.0576	587	20.6	-
Ĺa	(7)	`∢0`		(5)			40 L	٥٤	0.0124	0.0414	N/A	20.0	
Li	`s´	(8)		(5)			₹ÕĹ	«οί	0.0074	0.0246	N/A		
Mg	586	- 634	8.1	539	564	4.7	72	88	0.0003	0.0009	SáZ	8.5	
Mn	102	107	5.2	60	60	1.0	(0)		0.0010	0.0034	81	52.7	*
Ма	31	(35)	. –	27	(28)		·≪ÌŪ́	<ol< td=""><td>0.0128</td><td>0.0427</td><td>29</td><td>14.8</td><td></td></ol<>	0.0128	0.0427	29	14.8	
Na	115,962	120,108	3.6	121,337	120,622	0.5	194	202	0.0587	0.1957	118,650	4.5	
Жđ	(31)	40L		(27)			<ql< td=""><td><0L</td><td>0.0496</td><td>0.1653</td><td>N/A</td><td></td><td></td></ql<>	<0L	0.0496	0.1653	N/A		
ИÍ	17, 131	18,131	5.8	15,563	15,782	1.4	≺OL	<0L	0.0231	0.0768	16,347	9.6	
26	617	651	5.6	568	584	2.8	<0L	ØL	0.0831	0.2771	592	8.2	į
Re	(5)	⊲ا		(5)	<0L		حود	<0L	0.0173	0.0576	N/A		
Rh	<0L	<0L		<0L	<0L		<0Ľ	≪OL	0.0821	0.2738	N/A		
Ru	≪0L	<0L		<0F	<0L		≪OL	<0L	0.0350	0.1167	H/A		
Şb	43	<0L		37	₹0 L		<0L	∢0L	0.0475	0.1584	40	14.3	
Se	***	***		***	***		***	***	0.1515	0.5050	***		
Si	912	822	9.9	984	816		(34)		0.0549	0.1830	948	7.5	
Şr	431	443	2.9	410		1.2	Z	_2	0.0004	0.0013	421	4.8	
Te	۷0L	ØL.		40L	<ol< td=""><td></td><td>٩L</td><td>۹٦L</td><td>0.0950</td><td>0.3168</td><td>HZA</td><td></td><td></td></ol<>		٩L	۹٦L	0.0950	0.3168	HZA		
Th Ti	<0L 7	≪OL		<0Ļ	<0L		<ol< td=""><td><0£</td><td>0.0734</td><td>0.2448</td><td>N/A</td><td>• .</td><td></td></ol<>	<0£	0.0734	0.2448	N/A	• .	
71	√ 0Ĺ	<0L		6 √ 0L	(6)		<0 ل	<0L	0.0054	0.0179	7	9.5	
Ú.	15,389	15,802	2.7	14,508	0٤, 14,448	0.7	<0ξ	<0.L	0.549Z 0.5376	1.8308	N/A		
v	(3)	13,502 <0L		(2)	14,446 ≺0L	J.4	<0L	<0L	0.0090	1.7920	14,949	5.9	
Žn	207	269	29.7	188	244	7 0 T	(2)		0.0041	0.0301	N/A 198	9.5	
Zr	16	(17)	••	14	(14)		<0 Γ (5.1	<0Γ	0.0076	0.0253	15	13.8	
P	26,887	28,242	5.0	30,819	31,015	0.6	-0L	.ΩL	0.6133	2.0442	28,853	13.6	
81	- · • · · ·			,*	,				J		N/A		

Data, including calibration/QC, archived file (CP-325-601- 11/13/92

11/24/92

Note: 1) Values reliable to 2 1/2 significant digits. Bracketed results () are qualitative.
2) Blank is reported in ug/g "equivalence" to indicate blank effect on sample.
3) Sample results have not been adjusted for "blank" contribution.
4) At 50-100 times the 0.L., precision is estimated at +/-10% and accuracy at +/-15%.
5) Sample DL (ug/g) = (DL in ug/mL) * (ug/g Factor)
6) Off-line IEC: Results within 50% OL potentially bias high.
7) "*** flag: RPD > 20% and both sample results > 3*dL.
8) *** Selenium Channel non-functional.

Table 2-2j: SST Core 48, ICP Quarter Segment 1D, Bottom, Homogenization Test, Acid Digestion

ICP ANALYSIS REPORT -- Acid Digestion

PROJECT: SST PROCEDURE: PNL-ALO-211 Samo Log#: 1361-A1B 1361-A1B 0ilution: 1.00 5.00 1361-A2B 1361-A2B 1.00 5.00 5.00

Analyzed Date: 11/13/92

M&TE: WAS5672

ortucion:	1.00	3.00		1.00	5.00								
ug/g factor	200.5	1002.4 172		198.7	993.6								
(CP Run #	154	172		155	173				***Esti	na ted***	1361-A18		
										61	1721 470		
	Sample	Sample		Sample	Sample		8 Lank	Blank			Average		20%
	ug/g	ug/g	20if	u g/g	ug/g	2Dif	ug/g	' ug/g	ug/mL	ug/mL	ug/g		Flg
						• • • • •							
Ag	<0L			<0L	<0L				0.0078		N/A		
ĄŁ	9,451	9,348	1.1	8,838	8,964	1.4			0.1829	0.6096	9,144	6.7	
As	<0Ľ	≪OL.		«ەر	<0L				0.1014	0.3379	N/A		
В	87	(76)		6 2	<0L				0.0596	0.1987	75	34.2	
Ba .	52	50	5.7	47	47	0.5			0.0080	0.0267	75 50	10.1	
Be	40L	<0L		_ વદ	- 40℃				0.0032	0.0107	N/A		
Ca	17,274	18,552	7.4	15,626	17, 192	10.0			0.0016	0.0054	16,450	10.0	
čq	8	(8)		7	<0L				0.0075	0.0250	8	12.6	
Çe	<0L	-ØΓ		<0L	<0L				0.1019	0.3396	W / A		
Ça	<0L	.≪ΩL		<0L	<0L				0.1865	0.6217	N/A 233 14		
Cr	241	239	0.8	224	229	2.0			0.0113	0.0378	233	7.2	
Çu	14	⊲0 L		14	<0L				0.0088	0.0294	14	0.8	
Ōλ	<0L	<0L		<0L	≪OL				0.0053	0.0176	A/A		
Fe	17,755	17,652	0.6	16,752	17,088	2.0			0.0100	0.0334	17, 253	5.8	
K	649	(416)		614	(391)				0.3173	1.0576	632	5.4	
La	9	<0L		(7)	≺0 Ł				0.0124	0.0414	632 N/A 6 621 76		
Li	7	<0L		6	<0L				0.0074	0.0246	6	14.6	
Мg	651	666	2.2	591	627	6.2			0.0003	0.0009	621	9.7	
Mn	78	79	0.4	74	76	2.5			0.0010	0.0034	76	5.3	
Мо	33	(33)		31	(32)				0.0128	0.0427	32	5.9	
Нa	99,304	98,26 9	1.0	106,425	108,068	1.5			0.0587	0.1957	102,865	6.9	
Яф	39	<0L		(30)	<0L				0.0496	0.1653	A/K		
Жi	19,429	19,454	1.2	17,366	18,018	3.8			0.0231	0.0768	18.397	11.2	
Pb	723	725	0.3	645	665	3.0			0.0831	0.2771		11.4	
Re	(6)	≪OL		(6)	. <ol< td=""><td></td><td></td><td></td><td>0.0173</td><td>0.0576</td><td>N/A</td><td></td><td></td></ol<>				0.0173	0.0576	N/A		
Ŗħ	<ol< td=""><td><0L</td><td></td><td><0L</td><td><0L</td><td></td><td></td><td></td><td>0.0821</td><td>0.2738</td><td>N/A</td><td></td><td>*</td></ol<>	<0L		<0L	<0L				0.0821	0.2738	N/A		*
Ru	<0L	≪OL		<0L	⊲ود				0.0350	0.1167	N/A		
Şb	43	<0L		42	<0L				0.0475	0.1584	42	1.3	
Se	***	***	_	***	***				0.1515	0.5050	***		
Şi	1,290	1,164	7.8	974	866	11.1			0.0549	0.1830	1,132	28.0	
Şr	501	493	1.5	450	454	1.0			0.0004	0.0013	475	10.8	
Ţe	⊲OL	₹0 £		<0L	<ql< td=""><td></td><td></td><td></td><td>0.0950</td><td>0.3168</td><td>N/A</td><td></td><td></td></ql<>				0.0950	0.3168	N/A		
Th .	<0Ľ	<0L		<0L	≺OL				0.0734	0.2448	475 N/A N/A 7		
Ţ i		<0L		7	<0L				0.0054	0.0179	7	0.7	
Τt	<0L	<0∟		≪OL	< 0↓				0.5492	1.8308	N/A		
u	17,409	16,770	5.7	15,510	15,256	1.6			0.5376		16,460	11.5	
Ā	(2)	9,348 - OL 9,348 - OL 18,552 - (8) - OL 17,652 - (416) - OL 17,652 - (416) - OL - OL - OL - OL - OL - OL - OL - OL		(2)	<0L				0.0090				
Zn	235	267 14	.0	213	272	27.6			0.0041	0.0136	н/A 224	9.7	
25	(3)	<0L		14	(11)				0.0076	0.0253	A/A	•	
P	19,570	19,858 1	1.5	ZZ,926	23,634	3.1			0.6133	2.0442	21,248	15.8	
18										_	N/A		

Note: 1) Values reliable to 2 1/2 significant digits. Bracketed results () are qualitative.
2) Blank is reported in ug/g "equivalence" to indicate blank effect on sample.
3) Sample results have not been adjusted for "blank" contribution.
4) At 50-100 times the D.L., precision is estimated at +/-10% and accuracy at +/-15%.
5) Sample DL (ug/g) = (DL in ug/mL) * (ug/g Factor)
6) Off-line IEC: Results within 50% DL potentially bias high.
7) **** Salenium Channel non-functional.

Data, including calibration/QC, archived File 102-325-601- 11/13/92

11/24/92

Table 2-2k: SST Core 49, ICP Quarter Segment 1D, Top, Homogenization Test, Acid Digestion

ICP ANALYSIS REPORT -- Acid Digestion

PROJECT: PROCEDURE:		211		-					Analyzi		11/13/92 WA55672		
Samp Log#: Dilution: ug/g Factor (CP Run #	1.00	93- 1367-A1T 5.00 989.3 177		93- 1367-A2T 1.00 191.8 160	93- 1367-A2T 5.00 959.0 178		93- 1367-A3 1.00 194.8 158	93- 1367-A3 5.00 973.9 176		na ted***	1367-A1T		
*********	Sample ug/g	Sample ug/g	20 i i	Sample ug/g	Sampie ug/g	2011	Blank ug/g	8lank ug/g	OL ug/mL	QL Ug/mL	1367-AZT Average ug/g	RPO	20% Flg
Ag	<0L	≪JL		<0L	<0L		≪OL	<0L	0.0078	0.0261	·		
ΑĨ	39,402	39,492	0.7	46,588	46,756	0.4	≪0L	ØL	0.1829	0.6096	M/A	14 7	
As	<υΓ	<0L		≪OL	<01.	•••	<0L	₹OL	0.1014	0.3379	42,995	16.7	
8	101	(112)		42	⊲οι		77	(99)	0.0596	0.1987	N/A	97 1	
Ba	28	29	2.5	38	38	0.9	√OL	«οί	0.0080	0.0267	71 33	83.1 30.4	-
8e	حال	<ol< td=""><td></td><td>≺OL</td><td><ol< td=""><td></td><td>₹ŪL</td><td>≪OL</td><td>0.0032</td><td>0.0107</td><td>A/W</td><td>30.4</td><td>-</td></ol<></td></ol<>		≺OL	<ol< td=""><td></td><td>₹ŪL</td><td>≪OL</td><td>0.0032</td><td>0.0107</td><td>A/W</td><td>30.4</td><td>-</td></ol<>		₹ŪL	≪OL	0.0032	0.0107	A/W	30.4	-
Ca	14.646	15,951	8.9	19,372	21,496	11.0	489	821	0.0036	0.0054		27 0	_
Ċd	5	≪OL		7	(8)		خان	40 L	0.0075	0.0250	17,009	27.8 32.4	-
Ce	⊲≎L	≪OL		≪OL	<ÔL		٩٤	<0Ľ	0.1019	0.3396	N/A	34.4	
Co	≪OL	<0L		<ol< td=""><td>.≺0L</td><td></td><td>۵۱</td><td><0L</td><td>0.1865</td><td>0.6217</td><td>N/A</td><td></td><td></td></ol<>	.≺0L		۵۱	<0L	0.1865	0.6217	N/A		
Cr	168	174	3.3	219	223	1.8	«σι	<0L	0.0113	0.0378	194	26.2	
Cu	14	(12)		15	(14)		<0L	≪OL	0.0088	0.0294	15	11.8	
07	<0L	≪OL		<0L	<0L		<0L	<ol< td=""><td>0.0053</td><td>0.0176</td><td>N/A</td><td>11.0</td><td></td></ol<>	0.0053	0.0176	N/A	11.0	
F€	8,846	8,981	1.5	11,532	11,703	1.5	11	<0L	0.0100	0.0334	10, 189	26.4	•
K	367	(378)		525	(560)		حور	<0L	0.3173	1.0576	446	35.2	
La	9	(13)		10	(14)		<0 L	<0L	0.0124	0.0414	9	14.4	
Li	_(3)			(4)	(9)		٩OL	<0 L	0.0074	0.0246	N/Á		
Mg	389	405		494	523	5.9	70	92	0.0003	0.0009	441	23.9	•
Яn	48	50	3.5	60	61	2.7	(0)	ح0اد	0.0010	0.0034	54	21.3	•
Мо	30	(34)		38	43	13.0	«ÒĹ	اران	0.0128	0.0427	34	24.5	*
Na	115,843		0.7	86,588	86,879	0.3	218	214	0.0587	0.1957	101,215	28.9	
Nd	(22)			43	(73)		<0L	<0L	0.0496	0.1653	N/A		4
ИI	11,875	12,285	3.5	15,601	16,158	3.6	<0L	⊲uĹ	0.0231	0.0768	13,738	27.1	· 2
ዖቴ	485	504	4.0	670	682	1.8	<0L	≪OL	0.0831	0.2771	577	32.0	. 4
A	18.												

Яe

Rh

Ru

Se

Sí

S٢

Te

Th

Tí

Τī

Ü

Ÿ

Zn

Zr

(5)

<0L

<OL

(25)

716

297

(35)

<0L

<0L

9,708

32,278

(3)

(4)

8

170

<0L

<OL

<0L

(70)

649

299

<OL

<0L

<01. 9,984

<QL

(10)

33,361 3.4

217 28.1

(6)

0.5

2.8

- Note: 1) Values reliable to 2 1/2 significant digits. Bracketed results () are qualitative.
 2) Blank is reported in ug/g "equivalence" to indicate blank effect on sample.
 3) Sample results have not been adjusted for "blank" contribution.

<0L

42

710

406

(44)

<0L

<OL

(4)

221

13

13,416

18,582

4) At 50-100 times the D.L., precision is estimated at +/-10% and accuracy at +/-15%.
5) Sample Dt (ug/g) = (Dt in ug/mt) * (ug/g Factor)
6) Off-line IEC: Results within 50% Dt potentially bias high.
7) *** flag: RPD > 20% and both sample results > 3**qt.

<0L

<0L

<0L

(54)

406

<OL

<0L

(7)

13,517 0.8

273 23.7

40L

(16)

19,163 3.1

685 3.4

<0L

۷0L

<0L

<٥٢

37

<OL

<OL

<0L

<0L

(213)

<0L

(2)

<OL

<0L

0.0173

0.0821

0.0350

0.0475

0.0549

0.0004

0.0950

0.0734

0.0054

0.5492

0.5376

0.0090

0.0041

0.0076

0.6133

<0L

<0L

<ūt.

<0L

<OL

<0L

<OL

∕0L

ΦL

55

<OL

40L

0.0576

0.2738

0.1167

0.1584

0.5050

0.1830

0.0013

0.3168

0.2448

0.0179

1.8308

1.7920

0.0301

0.0136

0.0253

2.0442

8) *** Selenium Channel non-functional.

Oata, including calibration/QC, archived File ICP-325-601- 11/13/92

11/24/92

0.9

30.8 *

32.1 *

26.4 *

46.8

53.9

N/A

N/A

N/A

N/A

713

352

N/A

N/A

N/A

N/A

N/A

195

11

H/A

11,562

25,430

Table 2-21: SST Core 49, ICP Quarter Segment ID, Bottom, Homogenization Test, Acid Digestion

ICP ANALYSIS REPORT -- Acid Digestion

PROJECT: SST Analyzed Gate: 11/13/92. PROCEDURE: PNL-ALO-211 M&TE: WA55672 93- 93-93- 93-

Samp Log#: 1			1		1367-A28								
Dilution:	1.00	5.00		1.00	5.00								
ug/g factor				198.1	990.3								
ICP Run #	161	179		162	180					a Ced***	1367-A18		
									DL	QL	1367-A2B		
	Sample	Sample			Sample		Blank	8 l ank			Average		20%
	ug/g	ug/g	2D if	ug/g	ug/g	2Dif	ug/g	ug/g	ug/mL	ug/mL	ug/g	RPO	Flg
Ag	- વા	⊲OL		<0L	٩L					0.0261	N/A		
ĄĹ	41,757	42,056	0.7		47,875	2.3				0.6096	44,279	11.4	
As	<0L	<0L		<0L	<0L					0.3379			
8	47	≪or		45	≪0Ľ	0.5				0.1987		4.4	
8a	29		2.9	35	35	0.5				0.0267		17.5	
8e	- OL	<0L			<υ ι					0.0107			
Ca	16,856		9.4	18,738	21,051	12.3			0.0016	0.0054			
Çd	6	≪0L		7					0.0075			16.9	
Ça	<ql< td=""><td>≪OL</td><td></td><td><0L</td><td>≪OL.</td><td></td><td></td><td></td><td>0.1019</td><td>0.3396</td><td>N/A</td><td></td><td></td></ql<>	≪OL		<0L	≪OL.				0.1019	0.3396	N/A		
Co	<0L	<0L		∢0L	<0L					0.6217			
Č۲	190		3.2	207	216	4.3				0.0378		8.9	
Çu	13	(12)	l	17	(14)	1				0.0294	15	27.2	
DУ	<0L	≪OL		- OL	<0L					0.0176	N/A		
F e	9,050				11,544				0.0100	0.0334			*
K	450	(463)		518		•				1.0576	484	14.0	
Lạ	(7)			9	<0L					0.0414	H/A		
Li	(4)	(9)	٠	(5)						0.0246	H/A		
Mg	417	443	6.1	467						0.0009		11.2	
Mn	47	48	3.2	58		4.2				0.0034	52		•
Мо	32	(35)		36						0.0427		11.7	
Хa	87,982	88,699		80,067					0.0587	0.1957	84,025	9.4	
· Nd	35	(58)		42	(64)					0.1653		17.1	
Жí	12,912	13,399		14,838	15,651	5.5				0.0768	13,875		
Pb	508		0.0	616		2.1				0.2771	562	19.2	•
Яe	(7)) < 0L		(9)	, .O.C.					0.0576	H/A		
Rh	<0L	<0L		<0L	≪L					0.2738	N/A		
Ru	<0L	<0L		<0₺	<0L					0.1167	N/A		
\$5	37			42	≪OL					0.1584		13.5	
Se	***	***		***	***					0.5050	***	_	
Si	623		8.6	726		5.5				0.1830		15.3	
\$r	312		0.5	369		2.2				0.0013	340	16.8	
Te	(39)) ≪סנ		(46)						0.3168	N/A		
Th	<0L			· <0L	⊲oL					0.2448	N/A		
Ţi	(3)) (6)		(3)) ≺OL					0.0179	N/A		
71	<0L			<0L						1.8308			
U	10,027	10,31 <i>7</i>	2.9	12,114	12,262	1.2				1.7920		18.9	
٧	(3)			(4)						0.0301			
Zn	181	220	22.0	210	269	28.1			0.0041	0.0136	195	15.2	
Zr	10	(13))	13	(13)	1			0.0076	0.0253	11	24.6	
P	20,901	21,775	4.2	17,509	18,217	4.0			0.6133	2.0442	19,205	17.7	
Bi	-	•		•	-						A/K		

Note: 1) Values reliable to 2 1/2 significant digits. Bracketed results () are qualitative.
2) Blank is reported in ug/g "equivalence" to indicate blank effect on sample.
3) Sample results have not been adjusted for "blank" contribution.

Data, including calibration/QC, archived File ICP-325-601- 11/13/92

11/24/92

⁴⁾ At 50-100 times the D.L., precision is estimated at +/-10% and accuracy at */-15%.
5) Sample DL (ug/g) = (DL in ug/mL) * (ug/g Factor)
6) Off-line (EC: Results within 50% DL potentially bias high.
7) "** ftag: RPD > 20% and both sample results > 3*QL.

^{8) ***} Selenium Channel non-functional.

Table 2-3a: SST Core 47, ICP Core Composite Water Leach

ICP ANALYSIS REPORT -- Water Leach

PROJECT: SST PROCEDURE: PHL-ALO-211

Analyzed Oate:01/21/93 M&TE: WASS672

Samo Log#:	93-01358c1		9	3-01358c2		93	3-01358c3						
Oilutions	2.00			2.00			2.00						
ug/g factor	246.3			239.4			241.4						
ICP Run #	485			486			484				93-013586	-1	
, , , , , , ,									****		93-013586		
	Sample	Sample		Cupt.	Cupi.		Blank	3 Lank	٥L	QL	Average	•	20%
	ug/g	49/9	≫if	ug/g	D/Bn	2D if	49/9	ug/g	ug/mt_	ug/mi_	ug/g	200	Flg
							******			******	-3/3		
Ag	<0L			<0L			<0L		0.0081	0.0272	N/A		
AL	336			488			<ql< td=""><td></td><td>0.1904</td><td>0.5345</td><td>412</td><td>36.7</td><td></td></ql<>		0.1904	0.5345	412	36.7	
As	≪OL			<ol< td=""><td></td><td></td><td><0L</td><td></td><td>0.:055</td><td>0.3517</td><td>H/A</td><td>20.,</td><td></td></ol<>			<0L		0.:055	0.3517	H/A	20.,	
8	(18)			(21)			<0L		01.0620	0.2068	A/K		
Ba	⟨OL			₹0L			رة ا		0.0083	0.0278	A/K		
Se.	≪OL			⊲0L			<0L		0.0033	0.0112	N/A		
Ca	173			194			45		0.0017	0.0056	183	11.8	
ca	40 L			ابر. حول			<0L	,	0.0078	0.0260		11.4	
											A/K		
Ce	<0L			QL.			<0L		0.1060	0.3534	A/K		
Ca	<0L			-QL			<0L			0.5471	N/A		
Çr	173			176			«OL		0.0118	0.0393	174	1.5	
Cu	≪0 L			الان م			<0L		0.0092		A/K		
ĐΥ	<0L			≪OL			 ot 		0.0055	0.0183	A/K		
Fe	885			872			<0L		0.0104	0.0347	878	1.5	
ĸ	510			558			<0L		0.3302	1.1008	534	9.0	
Ļ	<0L			<0£			<0L		0.0129	0.0430	A/K		
Li	<0 L			<0₹			<ol,< td=""><td></td><td>0.0077</td><td>0.0256</td><td>A/K</td><td></td><td></td></ol,<>		0.0077	0.0256	A/K		
Нд	. 8			8			4		0.0003	0.0009	8	0.9	
λn	<0L			(0)			<0L		0.0011	0.0035	H/A		
Но	24			25			<ql< td=""><td></td><td>0.0133</td><td>0.0445</td><td>25</td><td>3.9</td><td></td></ql<>		0.0133	0.0445	25	3.9	
на	66,028			69,553			«o۴		0.0611	0.2037	67,790	5.2	
Hđ	< 0L			<0L			<0L		0.0516	0.1720	H/A		_
Жi	140			109			<0L		0.0240	0.0800	125	25.0	*
. РЪ	(41)			(55)			<0L		0.0865	0.2884	H/A		
Re	< 0↓			<0L			<0L		0.0180	0.0599	A/K		
Rh	٩OL			<0L			<0 L		0.0855	0.2850	N/A		
Ŕψ	<ol< td=""><td></td><td></td><td>√0L</td><td></td><td></td><td><ql< td=""><td></td><td>0.0364</td><td>0.1215</td><td>X/A</td><td></td><td></td></ql<></td></ol<>			√0 L			<ql< td=""><td></td><td>0.0364</td><td>0.1215</td><td>X/A</td><td></td><td></td></ql<>		0.0364	0.1215	X/A		
d2	40 L			<0L			<0L		0.0495	0.1649	H/A		
Se	<0L			<0L			<0L		0.1577	0.5256	H/A		
. Şi	112			122			<0L		0.0571	0.1904	117	7.7	
Ş٢	1			1			<ol< td=""><td></td><td>0.0004</td><td>0.0013</td><td>1</td><td>24.7</td><td>•</td></ol<>		0.0004	0.0013	1	24.7	•
Te	<0L			<0L			<0L		0.0989	0.3297	H/A		
Th	√0 L			<ql< td=""><td></td><td></td><td><0L</td><td></td><td>0.0764</td><td>0.2548</td><td>A/K</td><td></td><td></td></ql<>			<0L		0.0764	0.2548	A/K		
Ti	<0Ĺ			<0L			<0L		0.0056	0.0187	A/K		
Τι	<0L			<0L			<0L		0.5717	1.9056	A/A		
Ų	∢0L			(156)			<0L		0.5595	1.8651	N/A		
v	<0L			(2)			٠٥٤		0.0094	0.0313	X/A		
Žn	9			`9			9		0.0043	0.0142	~~9	0.4	
2	<0Ĺ			«σĹ			∢οĹ		0.0079	0.0263	ÄVK	***	
۶.	6,349			7,629			₹0 L		0.6383	2.1277	6,989	18.3	
Ši	4,447			. , 52, 9			`U.		0.0262		A/K	,	
• • • • • • • • • • • • • • • • • • • •											7/0		

Data, including calibration/GC, archived file [C2-325-601-01/21/93

01/26/93

Note: 1) Values reliable to 2 1/2 significant digits. Bracketed results () are qualitative.
2) Blank is reported in ug/g "equivalence" to indicate blank effect on sample.
3) Sample results have not been adjusted for "blank" contribution.
4) At 50-100 times the D.L., precision is estimated at +/-10% and accuracy at +/-15%.
5) Sample OL (ug/g) = (DL in ug/mL) = (ug/g Factor)
6) Off-line IEC: Results within 50% DL potentially bias high.
7) 20% "** flag: RPO > 20% and both sample results > 3°QL.

Table 2-3b: SST Core 48, ICP Core Composite Water Leach

ICP ANALYSIS REPORT -- Water Leach

93-01363c2

PROJECT: SST

PROCEDURE: PNL-ALO-211

Samp Log#: 93-01363c1

Analyzed Date:01/21/93 MATE: 4455672

Oilution: ug/g factor ICP Run #	2.00 229.9 487		,3	2.00 235.6 488					*******		93-01363; 93-01363;		
	Sample	Sample		Oupl.	Օսբե.		Blank	8 lank	ָם. סג	qL	Average	-4	20%
	ug/g	ug/g	#Dif	ug/g	ug/g	⊅if	ug/g	ug/g	ug/mL	ug/mL	ug/g	RPO	Fig
Ag	<0L			<0L					0.0081	0.0272	N/A		•••
AL	(115)			(104)					0.1904	0.6345	N/A		
As	<0L			₹0L					0.1055	0.3517	H/A		
8	84			90					0.0620	0.2068	87	4.8	
Вa	<ol< td=""><td></td><td></td><td><0L</td><td></td><td></td><td></td><td></td><td>0.0083</td><td>0.0278</td><td>H/A</td><td></td><td></td></ol<>			<0L					0.0083	0.0278	H/A		
8e	<0L			<0L					0.0033	0.0112	A/K		
Ça	59			60					0.0017	0.0056	60	0.8	
Çd	<0L			<0L					0.0078	0.0260	H/A		
Ce	<0L			<0L					0.1060	0.3534	H/A		
Ca	< 0↓			<0L					0.1941	0.6471	H/A		
Çr	217			234					0.0118	0.0393	225	7.6	
Cu	حە د			< 0 €					0.0092	0.0306	N/A		
0y	<0L			<0L					0.0055	0.0183	A/K		
Fe	1,128			1,149					0.0104	0.0347	1,139	1.9	
X	540			576					0.3302	1.1008	558	6.3	
La	<0L			<0L					0.0129	0.0430	H/A		
Ļi	<0L			40 ٤						0.0256	A/K		
Mg	7			7					0.0003	0.0009	7	0.0	
Mn	<0L			<0L					0.0011	0.0035	N/A		
Мо	29			31					0.0133	0.0445	30	6.1	
Nа	89,161			77,859					0.0611	0.2037	83,510	13.5	\$
жф	<۵۲			<0L					0.0516	0.1720	A/A		•
Ni	33			29					0.0248	0.0800	31	15.3	
۶۶	<0L			<0L					0.0865	0.2884	H/A		
Re	<0 L			٠OL					0.0180	0.0599	H/A		
Rh	≪OL.			<ol< td=""><td></td><td></td><td></td><td></td><td>0.0855</td><td>0.2850</td><td>H/A</td><td></td><td></td></ol<>					0.0855	0.2850	H/A		
Ru	۷OL			<0L					0.0364	0.1215	A/A		
\$b	«OL			<0L					0.0495	0.1649	A/A		
Se	<0L			<0L						0.5256	A/A		
\$ i	303			91			•		0.0571	0.1904		107:3	
Şr	1			1					0.0004	0.0013		15.1	
Te	«۵ ۲			<0L						0.3297	A/K		
Th	<0L			<0L					0.0764	0.2548	A/K		
Ti Tt	<0L			<ol< td=""><td></td><td></td><td></td><td></td><td></td><td>0.0187</td><td>A/K</td><td></td><td></td></ol<>						0.0187	A/K		
13	⟨ 0,			∢0L					U.5/17	1.9056	A/K		

Note: 1) Values reliable to 2 1/2 significant digits. Bracketed results () are qualitative.
2) Blank is reported in ug/g "equivalenca" to indicate blank effect on sample.
3) Sample results have not been adjusted for "blank" contribution.
4) At 50-100 times the D.L., precision is estimated at +/-10% and accuracy at +/-15%.
5) Sample DL (ug/g) = (DL in ug/mL) * (ug/g Factor)
6) Off-line IEC: Results within 50% OL potentially bias high.
7) 20% """ flag: RPD > 20% and both sample results > 3°CL.

<OL

<0L

5,461

<OL

<OL

8

<OL

11,918

V

Zn

Zr

Data, including calibration/QC, archived File [CP-325-601-01/21/93

01/26/73

A/K

N/A 9

N/A

H/A

7.9

8,690 74.3 *

0.0094 0.0313

0.0043 0.0142 0.0079 0.0263

0.6383 2.1277

1.8651

0.0263

0.5595

ICP ANALYSIS REPORT -- Water Leach

PROJECT: SST PROCEDURE: PHL-ALO-211

Analyzed Date:01/21/93 MATE: WASS672

									•				
Samo Log#:	93-0137161		93-0	11371c2									
Dilution:	2.00			5-00									
ug/g Factor				231.9									
CP Run #	489												
ICP XUN #	467			490							93-01371c		
									Esti	ಗಾ ೯ ಆಡೆ***	93-01371c	2	
	Sample	Sample		Oupl.	Oupt.		Blank	Blank	OL	αŁ	Average		20%
	ug/g	ug/g	2D if	ug/g	ug/g	2Dif	ug/g	ug/g	ug/mL	ug/mt	ug/g	RPO	fla
													4
PA PA	<0L			<0L					0.0081	0.0272	H/A		
ΑŨ	(114)			(96)					0.1904	0.6345	H/A		
As	≺OL			<0L						0.3517	N/A		
8	(21)			(23)					0.0620	0.2068			
Ša	<0 L			⟨OL						0.0278	N/A		
Se .	<0 L			<0L							N/A		
										0.0112	X/A		
Ca	89			66						0.0056	78	29.6	•
Cd	≪0 L			<0L						0.0260	A/K		
Ce	<0L			<0 L						0.3534	A/K		
Co	<0L			<ol< td=""><td></td><td></td><td></td><td></td><td>0.1941</td><td>0.6471</td><td>H/A</td><td></td><td></td></ol<>					0.1941	0.6471	H/A		
Cr	169			183					0.0118	0.0393	176	7.3	
Cu	<0L			<0L						0.0306	H/A		
Оy	<0L			<0L						0.0183	A/A		
Fe				944						0.0347	916	6.1	
K	428			468						1.1008			
Ĺa	≪OL			<0L							448	8.8	
L i	<0 L			<0L						0.0430	A/K		
									0.0077	0.0256	A/K		
Жд	6			- 6			•		0.0003	0.0009	á	3.4	
Mn				<ol< td=""><td></td><td></td><td></td><td></td><td></td><td>0.0035</td><td>H/A</td><td></td><td></td></ol<>						0.0035	H/A		
Но				25					0.0133	0:0445	24	10.6	
Ма	•			50,875					0.0611	0.2037	59,898	3.3	
Nd				<0L					0.0516	0.1720	A/A		
Иi	53			53					0.0240	0.0800	53	0.1	
ዖb	<0L			≺0L					0.0865	0.2884	N/A		
Яe	<0L			<0L						0.0599	A/K		
Rh	<0L			<0L					0.0855	0.2850	N/A		
Ru	<ol< td=""><td></td><td></td><td><0L</td><td></td><td></td><td></td><td></td><td>0.0364</td><td>0.1215</td><td>N/A</td><td></td><td></td></ol<>			<0L					0.0364	0.1215	N/A		
42				<0L					0.0495	0.1649	N/A		
Se	<0L			<ql< td=""><td></td><td></td><td></td><td></td><td>0.1577</td><td></td><td></td><td></td><td></td></ql<>					0.1577				
Şi	65			75							H/A	47.0	•
Şr	-			, 0					0.0571	0.1904	70	13.9	
Te									0.0004	0.0013	1	18.4	
				<0L						0.3297	N/A		
<u>Ih</u>	<0L			۷OL					0.0764	0.2548	A/R		
Ţί	≪O.L			<ol.< td=""><td></td><td></td><td></td><td></td><td>0.0056</td><td>0.0187</td><td>N/A</td><td></td><td></td></ol.<>					0.0056	0.0187	N/A		
T t	<0L			<0L					0.5717	1.9056	A/K		
u	<0L			<0L					0.5595	1.8651	N/A		
V	<0L			<0L					0.0094	0.0313	N/A		
Zn	8			6					0.0043	0.0142	7	27.1	
Zr	<0L			<0L					0.0079	0.0263	N/A		
P	4,422			3,900					0.6383	2.1277	4,161	12.5	
. 8 i	•			•						'	H/A	, 2.2	
											7/8		

Note: 1) Values reliable to 2 1/2 significant digits. Bracketed results () are qualitative.

Data, including calibration/QC, archived File ICP-325-601-

01/21/93

01/26/93

¹⁾ Values retracte to 2 1/2 significant digits. Bracketed results () are qualitative 2) Blank is reported in ug/g "equivalence" to indicate blank effect on sample.

3) Sample results have not been adjusted for "blank" contribution.

4) At 50-100 times the 0.L., precision is estimated at */-10% and accuracy at */-15%.

5) Sample OL (ug/g) = (Ot in ug/mL) = (ug/g factor)

6) Off-line IEC: Results within 50% OL potentially bias high.

7) 20% """ flag: RPO > 20% and both sample results > 3°OL.

93-01372g

Table 2-3d: SST C-109, ICP Hot-Cell Blank, Water Leach

ICP ANALYSIS REPORT -- Water Leach

93-01327g2

PROJECT: SST

PROCEDURE: PNL-ALO-211

Samp Log#: 93-01327g1

Analyzed Date:01/22/93

M&TE: WA55672

Dilution: ug/mL Fctr	1.00			1.00			1.00							
ICP Run #	498			502			500				93-01327g1			
									Esti	ma ced	93-0132792			
	Sample	Sample	YD LE	Dupl.	Oupl.	wn i z	8 Lank	Blank	DL	QL	Average		20%	
	ug/mL	ug/mL	20 if	ug/mL	ug/mL	2Dif	ug/mL	ug/mL	ug/mL	ug/mL	ug/mL	KPO	Flg	
Ag	<0L			<0L			⊲0L		0.0081	0.0272	N/A			
Αĺ	<0L			<0L			< 0L		0.1904	0.6345	N/A			
As	<0L			<0L			<0L		0.1055	0.3517	N/A			
8	<0L			≺OL			<0L		0.0620	0.2068	N/A			
Ba	<0L			<0L			<0L		0.0083	0.0278	H/A			
8e	<0L_			<0L_			<0L		0.0033	0.0112	N/A			
Ca	0.637			0.637			0.079		0.0017	0.0056	0.637	0.1		
Çđ	<dl< td=""><td></td><td></td><td><0L</td><td></td><td></td><td><0L</td><td></td><td>0.0078</td><td>0.0260</td><td>N/A</td><td></td><td></td><td></td></dl<>			<0L			<0L		0.0078	0.0260	N/A			
Çe	<ol< td=""><td></td><td></td><td><0L</td><td></td><td></td><td><0L</td><td></td><td>0.1060</td><td>0.3534</td><td>N/A</td><td></td><td></td><td></td></ol<>			<0L			<0L		0.1060	0.3534	N/A			
Co	<0L			<0L			<0L		0.1941	0.6471	N/A			
Çr	(0.016)			(0.016)			<0L		0.0118	0.0393	N/A			
Çu	<0L			<0L			<0L		0.0092	0.0306	N/A			
Dy	<0L			<0L			<0L		0.0055	0.0183	N/A			
Fe	0.156			0.169			<0L		0.0104	0.0347	0.162	7.6		
K	(0.365)			<0L			<0L	•	0.3302	1.1008	N/A			
La	<0L			<0L			<0L		0.0129	0.0430	N/A			
Ļi	<0L			≪OL			<0L		0.0077	0.0256	N/A			
Mg	0.056			0.056			0.004		0.0003	0.0009	0.056			
Mn	0.007			0.006			<0L		0.0011	0.0035	0.006	7.3		
Мо	<0L_			≪0L_			<0L		0.0133	0.0445	N/A			į
Ma	9.467			9.463			0.381		0.0611	0.2037	9.465	0.0		
Nd	<0L			<0L			ح0L		0.0516	0.1720	H/A			÷,
Ni	0.110			0.108			<0L		0.0240	0.0800	0.109	2.0		
P6	<0L			٩L			<0L		0.0865	0.2884	N/A			
Re	<0L			٩L			<0L		0.0180	0.0599	N/A			
Rh	<0L			<0L			<0L		0.0855	0.2850	N/A			
Ru	<0L			<0L			<0L		0.0364	0.1215	N/A			
Sb	<0L			<0L			<0L		0.0495	0.1649	N/A			
. Se	<0L 0.796			<0L 0.784			<0L		0.1577	0.5256	H/A			
Si Sr	0.798			0.784			<ol (0.000)</ol 		0.0571	0.1904	0.790	1.5		
Te	<0L			<0L					0.0004	0.0013	0.007	0.2		
Th	<0L			<0L			<0₺ <0₺		0.0989	0.3297	N/A			
Tí	(0.007)			<0£			(0.007)		0.0764 0.0056	0.2548	N/A			
Ti	(0.007) <0L			<0L						0.0187	N/A			
Ü	(0.751)			(0.598)			<0L		0.5717	1.9056	N/A			
V							<0L		0.5595 0.0094	1.8651	N/A			
Zn	<ol 0.016</ol 			<0L 0.016			<ol 0.018</ol 		0.0043	0.0313	N/A	1 0		
Zr	0.016 <0L			<0L			0.01a <0L		0.0043	0.0142 0.0263	0.016	1.9		
P	(0.651)			<0L					0.6383		N/A			
e Bi	(0.651)			₩.			<0L		0.0303	2.1277	N/A			
51											H/A			

Note: 1) Values reliable to 2 1/2 significant digits. Bracketed results () are qualitative.

2) Blank is reported in ug/g "equivalence" to indicate blank effect on sample.

3) Sample results have not been adjusted for "blank" contribution.

4) At 50-100 times the D.L., precision is estimated at +/-10% and accuracy at +/-15%.

5) Sample DL (ug/mL) = (DL in ug/mL) * (ug/mL fctr)

6) Off-line IEC: Results within 50% DL potentially bias high.

7) 20% "*" flag: RPD > 20% and both sample results > 3*QL.

Data, including calibration/QC, archived File ICP-325-601-01/22/93

Segments,

**** ION CHROMATOGRAPHY REPORT ****

FLUORIDE

SAMPLE VALUES ARE IN ug/g

TANK C-109, CORE 47 TO 49

	T	ALUES AH	L IIV ug/g		I			-109, CORE	
	SMPL	DUP		BLANK	< SAN	IPLE SPIK	E>	SPIKE CO	NTROL
SAMPLE ID	1	2	RPD	3	4	EXPECT	% REC	5	% REC
93-01355/C47-1B	300	300	0%	< 10	500	310	65%	340	136%
93-01356/C47-1C	300	300	0%						
93-01357/C47-1D	300	300	0%						*
93-01360/C48-1C	500	500	0%		•				
93-01361/C48-1D	1100	400	93%	< 10	3500	260	***	330	132%
93-01365/C49-1B	< 300	< 300							
93-01366/C49-1C	300	300	0%		500	220	91%		
93-01367/C49-1D	1000	900	11%						
93-01358/C47CMP	400	400	0%	< 30			-	· · · · · · · · · · · · · · · · · · ·	
93-01363/C48CMP	2200	400	138%					· · · · · · · · · · · · · · · · · · ·	
93-01371/C49CMP	400	400	0%					1 1	
93-01354/DL	< 200	< 200		< 30	300	40	750%	330	132%
			1	, , <u>, , , , , , , , , , , , , , , , , </u>				TRUE= 250) ua/mi

- 1. THIS DATA SUMMARY SHEET INFORMATION IS NOT COMPLETE WITHOUT THE RUN NARRATIVE.
- 2. Nomenclature used for this data set: 1 = Sample; 2 = Duplicate; 3 = Blank; 4 = Sample spike; 5 = Spike Control.
- 3. Calculate sample values; analyte in sample extract x (sample wt. + diluent wt.)/ sample wt.
- 4. Calculate spike recovery: ("4" "1")/"EXPECT" or, "4"/"EXPECT" if "1" < sample det. lim.; "***" indicates "1"/"EXPECT" > 4.
- 5. Calculate spike control recovery: (spike control) / true value for analyte in stock spike.

Table

CHLORIDE

SAMPLE VALUES ARE I	IN	ua/a
---------------------	----	------

TANK C-109, CORE 47 TO 49

< SAM 4 800	EXPECT 310	E> % REC 97%	· · · · · · · · · · · · · · · · · · ·	% REC
800				
	310	97%	240	96%
1100				
1100				
1100				
1100				
	260	38%	240	96%
		3.72.		
1000	220	91%		
1300	40	***	230	92%
1300	_	40	40 ***	
	1300	1300 40	1300 40 ***	1300 40 *** 230 TRUE = 250

- 1. THIS DATA SUMMARY SHEET INFORMATION IS NOT COMPLETE WITHOUT THE RUN NARRATIVE.
- 2. Nomenclature used for this data set: 1 = Sample; 2 = Duplicate; 3 = Blank; 4 = Sample spike; 5 = Spike Control.
- 3. Calculate sample values; analyte in sample extract x (sample wt. + diluent wt.)/ sample wt.
- 4. Calculate spike recovery: ("4" "1")/"EXPECT" or, "4"/"EXPECT" if "1" < sample det. tim.; "***" indicates "1"/"EXPECT" > 4.
- 5. Calculate spike control recovery: (spike control) / true value for analyte in stock spike.

NITRITE

SAMPLE VALUES ARE IN ug/g

TANK C-109, CORE 47 TO 49

	SMPL	DUP	1	BLANK	SAN	IPLE SPIK	F>	SPIKE CO	NTROL	
SAMPLE ID	1		RPD	3	4	EXPECT	% REC	5	% REC	
93-01355/C47-1B	27000	28800	6%	< 20	41700	18300	80%	12200	81%	
93-01356/C47-1C	37000	37000	0%							
93-01357/C47-1D	40000	39000	3%							
93-01360/C48-1C	49000	53000	8%							
93-01361/C48-1D	49000	50000	2%	< 20	58000	15600	58%	12100	81%	
93-01365/C49-1B	25800	27100	5%							
93-01366/C49-1C	42000	45000	7%		56000	13300	105%			
93-01367/C49-1D	46000	44000	4%							
93-01358/C47CMP	38000	40000	5%	< 60						
93-01363/C48CMP	42000	48000	13%							
93-01371/C49CMP	38000	39000	3%				:			
93-01354/DL	71000	71000	0%	< 50	74000	2300	***	11500	77%	
	<u> </u>			<u> </u>	<u> </u>	<u> </u>	<u> </u>	TRUE= 150	00 uz (m)	

TRUE = 15000 ug/mL

- 1. THIS DATA SUMMARY SHEET INFORMATION IS NOT COMPLETE WITHOUT THE RUN NARRATIVE.
- 2. Nomenclature used for this data set: 1 = Sample; 2 = Duplicate; 3 = Blank; 4 = Sample spike; 5 = Spike Control.
- 3. Calculate sample values: analyte in sample extract x (sample wt. + diluent wt.)/ sample wt.
- 4. Calculate spike recovery: ("4" "1")/"EXPECT" or, "4"/"EXPECT" if "1" < sample det. ilm.; "***" indicates "1"/"EXPECT" > 4.
- 5. Calculate spike control recovery: (spike control) / true value for analyte in stock spike.

Table

NITRATE

SAMPL	E VAL	UES A	RE	IN :	ua/a
-------	-------	-------	----	------	------

TANK	C-109.	CORE	47	TO	49
------	--------	------	----	----	----

	TANK C-109, CORE 47 10									
	SMPL	DUP	·	BLANK	< SAN	PLE SPIK	E>	SPIKE CO	NTROL	
SAMPLE ID	1 1	2 1	RPD	3	4	EXPECT	% REC	5	% REC	
93-01355/C47-1B	26900	28300	5%	< 20	56500	24400	121%	26200	1319	
93-01356/C4 7 -1C	36000	36000	0%							
93-01357/C47-1D	39000	38000	3%							
93-01360/C48-1C	55000	57000	4%							
93-01361/C48-1D	52000	55000	6%	< 20	78000	20700	126%	26600	1339	
93-01365/C49-1B	25200	26200	4%							
93-01366/C49-1C	40000	44000	10%		68000	17800	157%			
93-01367/C49-1D	44000	42000	5%							
93-01358/C47CMP	37000	37000	0%	< 60						
93-01363/C48CMP	45000	51000	13%							
93-01371/C49CMP	35000	37000	6%							
93-01354/DL	72000	72000	0%	< 50	79000	3100	***	27500	138%	

TRUE = 20000 ug/mL

- 1. THIS DATA SUMMARY SHEET INFORMATION IS NOT COMPLETE WITHOUT THE RUN NARRATIVE.
- 2. Nomenclature used for this data set: 1 = Sample; 2 = Duplicate; 3 = Blank; 4 = Sample spike; 5 = Spike Control.
- 3. Calculate sample values: analyte in sample extract x (sample wt. + diluent wt.)/ sample wt.
- 4. Calculate spike recovery: ("4" "1")/"EXPECT" or, "4"/"EXPECT" If "1" < sample det. lim.; "***" indicates "1"/"EXPECT" > 4.
- 5. Calculate spike control recovery: (spike control) / true value for analyte in stock spike.

Table

2

*** ION CHROMATOGRAPHY REPORT

PHOSPHATE

SAMPLE VALUES ARE IN ug/g

TANK C-109, CORE 47 TO 49

SAMPLE ID 1 2 RPD 3 4 EXPECT % REC 5 % REC 93-01355/C47-1B 7100 7500 5% < 20 11300 4270 98% 3500 100 93-01356/C47-1C 9600 9500 1%		SAMPLE VALUES ARE IN UG/G						TANK G-109, COHE 47 10 4						
93-01355/C47-1B 7100 7500 5% < 20 11300 4270 98% 3500 100 93-01356/C47-1C 9600 9500 1% 93-01356/C47-1D 34000 55000 47% 93-01360/C48-1C 15000 16500 10% 93-01361/C48-1D 38000 34000 11% < 20 61000 3600 *** 3500 100 93-01365/C49-1B 6000 6200 3% 93-01366/C49-1C 8900 8700 2% 11200 3110 74% 93-01366/C49-1D 24300 26000 7% 93-01358/C47CMP 20100 24000 18% < 60 93-01358/C48CMP 35900 17500 69% 93-01361/C49CMP 13500 12000 12% 93-01354/DL 13500 13500 0% < 50 14300 540 *** 3500 100		SMPL DUP			BLANK	< SAN	IPLE SPIK	SPIKE CONTROL						
93-01356/C47-1C 9600 9500 1% 93-01357/C47-1D 34000 55000 47% 93-01360/C48-1C 15000 16500 10% 93-01361/C48-1D 38000 34000 11% < 20 61000 3600 *** 3500 100 93-01365/C49-1B 6000 6200 3% 93-01366/C49-1C 8900 8700 2% 11200 3110 74% 93-01367/C49-1D 24300 26000 7% 93-01358/C47CMP 20100 24000 18% < 60 93-01363/C48CMP 35900 17500 69% 93-01371/C49CMP 13500 12000 12% 93-01354/DL 13500 13500 0% < 50 14300 540 *** 3500 100	SAMPLE ID	1	2	RPD	3	4	EXPECT	% REC	5	% REC				
93-01357/C47-1D 34000 55000 47% 93-01360/C48-1C 15000 16500 10% 93-01361/C48-1D 38000 34000 11% < 20 61000 3600 *** 3500 100 93-01365/C49-1B 6000 6200 3% 93-01366/C49-1C 8900 8700 2% 11200 3110 74% 93-01367/C49-1D 24300 26000 7% 93-01358/C47CMP 20100 24000 18% < 60 93-01363/C48CMP 35900 17500 69% 93-01371/C49CMP 13500 12000 12% 93-01354/DL 13500 13500 0% < 50 14300 540 *** 3500 100	93-01355/C47-1B	7100	7500	5%	< 20	11300	4270	98%	3500	1009				
93-01360/C48-1C 15000 16500 10% 93-01361/C48-1D 38000 34000 11% < 20 61000 3600 *** 3500 100 93-01365/C49-1B 6000 6200 3% 93-01366/C49-1C 8900 8700 2% 11200 3110 74% 93-01367/C49-1D 24300 26000 7% 93-01358/C47CMP 20100 24000 18% < 60 93-01363/C48CMP 35900 17500 69% 93-01371/C49CMP 13500 12000 12% 93-01354/DL 13500 13500 0% < 50 14300 540 *** 3500 100	93-01356/C47-1C	9600	9500	1%										
93-01361/C48-1D	93-01357/C47-1D	34000	55000	47%										
93-01365/C49-1B 6000 6200 3% 93-01366/C49-1C 8900 8700 2% 11200 3110 74% 93-01367/C49-1D 24300 26000 7% 93-01358/C47CMP 20100 24000 18% < 60 93-01363/C48CMP 35900 17500 69% 93-01371/C49CMP 13500 12000 12% 93-01354/DL 13500 13500 0% < 50 14300 540 *** 3500 100	93-01360/C48-1C	15000	16500	10%										
93-01366/C49-1C 8900 8700 2% 11200 3110 74% 93-01367/C49-1D 24300 26000 7% 93-01358/C47CMP 20100 24000 18% < 60 93-01363/C48CMP 35900 17500 69% 93-01371/C49CMP 13500 12000 12% 93-01354/DL 13500 13500 0% < 50 14300 540 *** 3500 100	93-01361/C48-1D	38000	34000	11%	< 20	61000	3600	***	3500	1009				
93-01367/C49-1D 24300 26000 7% 93-01358/C47CMP 20100 24000 18% < 60 93-01363/C48CMP 35900 17500 69% 93-01371/C49CMP 13500 12000 12% 93-01354/DL 13500 13500 0% < 50 14300 540 *** 3500 100	93-01365/C49-1B	6000	6200	3%										
93-01358/C47CMP 20100 24000 18% < 60 93-01363/C48CMP 35900 17500 69% 93-01371/C49CMP 13500 12000 12% 93-01354/DL 13500 13500 0% < 50 14300 540 *** 3500 100	93-01366/C49-1C	8900	8700	2%		11200	3110	74%						
93-01363/C48CMP 35900 17500 69% 93-01371/C49CMP 13500 12000 12% 93-01354/DL 13500 13500 0% < 50 14300 540 *** 3500 100	93-01367/C49-1D	24300	26000	7%										
93-01371/C49CMP 13500 12000 12% 93-01354/DL 13500 13500 0% < 50 14300 540 *** 3500 100	93-01358/C47CMP	20100	24000	18%	< 60									
93-01354/DL 13500 13500 0% < 50 14300 540 *** 3500 100	93-01363/C48CMP	35900	17500	69%										
100	93-01371/C49CMP	13500	12000	12%										
	93-01354/DL	13500	13500	0%	< 50	14300	540	***	3500	1009				
TOUT OF OR ALL														

TRUE= 3500 ug/mL

- 1. THIS DATA SUMMARY SHEET INFORMATION IS NOT COMPLETE WITHOUT THE RUN NARRATIVE.
- 2. Nomenclature used for this data set: 1 = Sample; 2 = Duplicate; 3 = Blank; 4 = Sample spike; 5 = Spike Control.
- 3. Calculate sample values: analyte in sample extract x (sample wt. + diluent wt.)/ sample wt.
- 4. Calculate spike recovery: ("4" "1")/"EXPECT" or, "4"/"EXPECT" if "1" < sample det. lim.; """" indicates "1"/"EXPECT" > 4.
- 5. Calculate spike control recovery: (spike control) / true value for analyte in stock spike.

and

ION CHROMATOGRAPHY REPORT ****

SULFATE

	SAMPLE V	ALUES AR	E IN ug/g]		TANK C-109, CORE 47 TO 4				
	SMPL	DUP	BLANK < SAMPLE SPIKE> SPIKE			SPIKE CO	NTROL			
SAMPLE ID	1	2	RPD	3	4	EXPECT	% REC	5	% REC	
93-01355/C4 7 -1B	4900	5200	6%	40	9700	4580	105%	4000	107%	
93-01356/C47-1C	7100	7100	0%							
93-01357/C47-1D	7600	7100	7%		:					
93-01360/C48-1C	10800	11200	4%							
93-01361/C48-1D	10000	10000	0%	- 40	13000	3900	77%	4000	107%	
93-01365/C49-1B	4500	4800	6%							
93-01366/C49-1C	7900	8400	6%		11900	3340	120%			
93-01367/C49-1D	7900	8300	5%							
93-01358/C47CMP	7200	7400	3%	70						
93-01363/C48CMP	8900	9600	8%	` 						
93-01371/C49CMP	6200	6900	11%							
93-01354/DL	12800	12800	0%	< 50	13700	580	***	3900	104%	

TRUE = 3750 ug/mL

- 1. THIS DATA SUMMARY SHEET INFORMATION IS NOT COMPLETE WITHOUT THE RUN NARRATIVE.
- 2. Nomenclature used for this data set: 1 = Sample; 2 = Duplicate; 3 = Blank; 4 = Sample spike; 5 = Spike Control.
- 3. Calculate sample values: analyte in sample extract x (sample wt. + diluent wt.)/ sample wt.
- 4. Calculate spike recovery: ("4" "1")/"EXPECT" or, "4"/"EXPECT" if "1" < sample det. lim.; "***" indicates "1"/"EXPECT" > 4.
- 5. Calculate spike control recovery: (spike control) / true value for analyte in stock spike.

2-45

ION CHROMATOGRAPHY REPORT ****

FREE CN

SAMPL	E 1/A1	HEC	ADE	IN uala
SAMPL	.E VAL	LUES	AHE.	IN UQ/O

TANK	C-109,	CORE	47 TO 49
------	--------	------	----------

	SAMPLE V	ALULU AII			1ANK C-103, CORE 47 10 4					
	SMPL DUP			BLANK	< SAMPLE SPIKE>			SPIKE CONTROL		
SAMPLE ID	1	2	RPD	3	4	EXPECT	% REC	5	% REC	
93-01355/C47-1B	550	590	7%	< 0.2						
93-01356/C47-1C	840	812	3%					:		
93-013 57/ C4 7 -1D	910	900	1%		•					
93-01360/C48-1C	1473	1478	0%							
93-01361/C48-1D	1350	1370	1%	< 0.2						
93-01365/C49-1B	350	380	8%							
93-01366/C49-1C	620	670	8%				<u></u>			
93-01367/C49-1D	700	730	4%							
93-01358/C47CMP	820	810	1%	< 1.2						
93-01363/C48CMP	1230	1320	7%							
93-01371/C49CMP	540	560	4%							
93-01354/DL	1320	1350	2%	< 0.5			1			

THERE ARE NO SPIKES OR SPIKE CONTROLS FOR THIS SET OF DATA.

NOTES:

- 1. THIS DATA SUMMARY SHEET INFORMATION IS NOT COMPLETE WITHOUT THE RUN NARRATIVE.
- 2. Nomenclature used for this data set: 1 = Sample; 2 = Duplicate; 3 = Blank; 4 = Sample spike; 5 = Spike Control.
- 3. Calculate sample values; analyte in sample extract x (sample wt. + diluent wt.)/ sample wt.
- 4. Calculate spike recovery: ("4" "1")/"EXPECT" or, "4"/"EXPECT" if "1" < sample det. llm.; "**** indicates "1"/"EXPECT" > 4.

-

5. Calculate spike control recovery; (spike control) / true value for analyte in stock spike.

Table 2-4h: SST C-109, Hot-Cell Blank, IC

ION Chromatography Report

Simple values are in $\mu g/ml$.

SAMPLE ID	F	CL	NO ₂	NO,	PO ₄	SO ₄	CN
93-01327-N1, Sample	<0.5	0.5	2.3	7	2	2	0.14
93-01327-N2, Duplicate	<0.5	0.5	2.3	7	2	2	0.15
93-01372-N1, DIW Blank	<0.5	<0.5	<1	<1	<1	<1	<0.05



Table 2-5: SST Cores 47, 48, and 49 Core Composites GFAA Results

Lab ID# 93-01358-A1/B1 -A2/B2 -A3/B3		Sb <u>μg/g</u> <3.1 <3.2 <0.6	As <u>μg/g</u> 73 89 <0.5	Se <u>μg/g</u> <2.4 <2.4 <0.3
93-01363-A1/B1 -A2/B2	Core 48 Composite Duplicate	<0.6 <0.6	1.4	<2.4 <2.5
93-01371-A1/B1 -A2/B2 -A3/B3	Core 49 Composite Duplicate Process Blank	<2.9 <2.9 <0.6	118 110 1.0	<2.3 <2.3 <0.3
93-01327-G1	Hot Cell Blank	nd	<u>ид/L</u> <2.2	<u>μg/L</u> <1.2
93-01372-G	DIW Blank	nd	<2.2	<1.2
Lab ID#	OC Sample ID	%Rec	%Rec	%Rec
93-01358-A4/B4 -A5/B5	Core 47 Spiked Sample Spike Blank	n/a 93%	n/a 102%	n/a 84% å
93-01363-A4/B4	Core 48 Spiked Sample	71%	72%	n/a
93-01371-A4/B4 -A5/B5	Core 49 Spiked Sample Spike Blank	n/a 91%	n/a 98%	n/a 87%

Notes:

Sample designator = "A" for Sb and "B" for As and Se. Process Blank: μ g/g results adjusted for sample dilution factors. "n/a" = spike less than 25% of sample result or diluted below IDL. nd = not determined

Table 2-6: SST Cores 47, 48, and 49 Core Composite Hg, Cr(VI), and NH_3-N Inorganic Results

MERCURY: Core Composites, Liquid Composite, and Cell Blank

						** Recovery**
		******	Results ***	***		Spike Spike
		<u>Sample</u>	Dupl.	Blank	<u>RPD</u>	Sample Control
Core 47 Composite				<u>-</u>		<u> </u>
93-01358-D1-D5	(μg/g)	8.5	9.2	<0.008	8.2%	(a) 128%
Core 48 Composite	•					
93-01363-D1/D2/D4	(μg/g)	6.5	6.6		2.2%	(a)
Core 49 Composite						
93-01371-D1/D2/D4	(μg/g)	6.5	6.8		4.2%	(a)
Liquid Composite						
93-01354-N1-N5	$(\mu g/g)$	0.090	0.092	< 0.001	1.4%	129% 96%
	(μg/L)	110	112	< 0.6		
Cell Blank						
93-01327-D1-D3	(μg/L)	< 0.3	< 0.3	< 0.3		(b)

Chromium (VI) and Ammonia (NH₃-N): Core Composites

	******	****** Results *****				
	<u>Sample</u>	Dupl.	Blank	RPD		
Core 47 Composite						
93-01358-C1-C3 Cr(VI) (μ	ıg/g) 48	46	<5	4%		
, , ,	1g/g) 44	43	<5	3%		
Core 48 Composite						
93-01363-C1-C2 Cr(VI) (μ	1g/g) 36	37		3%		
•	ıg/g) 64	58		11%		
Core 49 Composite						
93-01371-C1-C2 Cr(VI) (μ	ıg/g) 29	60		70% (c)		
Cr(VI) reru	in 27					
NH ₃ -N (µ	1g/g) 52	57		8%		

Notes:

- (a) Hg spike <<25% of sample Hg concentration; spike unrecoverable.
- (b) Since both Hg results are less than Detection Limit, RPD is N/A.
- (c) Reason for high RPD is unknown; insufficient sample to rerun Duplicate.

Table 2-7: SST Cores 47, 48, and 49, TOC/TIC/TC Results

	ct - Quarter Segments	<u>ΤΟC (μg/g)</u>	IIC (μg/g)	<u>ΤC (μg/g)</u>
Core 1B		2200 2100 127% Recov	5500 5300 87% Recov	7800 7400
10	93-01356-J1	2000	5000	7100
	-J2	2000	5400	7300
1D	93-01357-J1	2200	5300	7500
	-J2	2200	5400	7700
Core	48			
	93-01360-J1	3500	8300	12000
	-J2	3800	9000	13000
10	93-01361-J1 -J2 -J3 Method Blk -J4 Spiked Spl	3800 3200 99 111% Recov	7100 7800 70 96% Recov	11000 11000 170
1D	93-01361-Jl (repeat)	3000	6500	9500
	-J2 (repeat)	3100	6300	9400
	-J3 (repeat)	16	6	22
Core	49		,	
18	93-01365-J1 -J2 -J4 Spiked Spl	1700 1900 118% Recov	3900 3900 120% Recov	5600 5800
10	93-01366-J1	2100	6500	8600
	-J2	2300	6700	9000
10	93-01367-J1	2600	6600	9200
	-J2	2500	7000	9600
	-J3 Method B1k	54	17	71

Table 2-7: SST Cores 47, 48, and 49, TOC/TIC/TC Results, cont'd

Direct - Core Composites	<u>ΤΟC (μg/g)</u>	TIC (μg/g)	TC (μg/g)
Core 47 Composite 93-01358-J1 -J2 -J3 Method Blk	3000 3300 < 50	5800 5800 < 50	8800 9100 · < 50
Core 48 Composite 93-01363-J1 -J2	2900 3000	5000 5300	7900 8300
Core 49 Composite 93-01371-J1 -J2 -J3 Method Blk	2100 2800 < 50	5100 5700 53	7200 8500 53
<u>Water Leach - Core Composites</u> Core 47 Composite			
93-01358-C1 -C2 -C3 Method Blk	2460 . 2150 90	5420 5990 150	7880 8140 240
Core 48 Composite 93-01363-C1 -C2	3330 2830	5520 5770	8850 8600
Core 49 Composite 93-01371-C1 -C2	2210 2440	4540 4290	6750 6730
Liquid Composite (Water Leach) 93-01354-C1 -C2 -C3 Method Blk	2500 2700 < 10	6300 6000 < 10	8800 8700 < 10
Hot Cell Blank	TOC (μg/ml)	TIC (μq/ml)	<u>TC (μg/ml)</u>
93-01327 93-01372 DIW Blank	3 1	2 < 0.5	5 1

Table 3-1a: SST Cores 47, 48, and 49, Gamma Energy Analysis (GEA) Results

Sample ID	*	Co(60) <i>µ</i> ci/g	+/- % error	Cs(137) #ci/g	+/- % error	Eu(152) <i>μ</i> ci/g	+/-% error	Eu(154) μci/g	+/- % error	Eu(155) μci/g	+/- % error	Am(241) μci/g	+/- % error
Tank C-109 Lie	quid Co	nposite											
*93-01354-N-1	A/smp	1.46E-03	5	5.60E00	4			<2.8E-04		<2.4E-03		<1.4E-03	
*93-01354-N-2			6	5.62E00	4			<3.2E-04		<2.4E-03		<1.4E-03	
*93-01354-N-3				<2E-05				<8.7E-06		<6.0E-06		<3.5E~06	
*93-03290-N	DIM	<4E-06		2.94E-05	17			<7.5E-06		<7.7E-06		<4.4E-06	
Tank C-109, Co Core 47	ore Com	oosite, Wat	er Łeac	h									
93-01358-C-1	W/omn	7 275 04	17	0.035.00									
			17	9.07E+00	4			<2.0E-03		<8.0E-03		<3.9E-03	
93-01358-C-2			19	9.40E+00	4			<2.4E-03		<8.1E-03		<4.0E-03	
93-01358-C-3 Core 48				4.67E-03	7			<7.7E-04		<5.2E-04		<2.2E-04	
93-01363-C-1			14	7.95E+00	4			<7.6E-04		<7.0E-03		<3.3E-03	
93-01363-C-2	W/dup	1.10E-03	13	1.07E+01	4			<5.5E-04		<8.1E-03		<3.9E-03	
Core 49													
93-01371-C-1			22	5.61E+00	4			<1.7E-03		<6.1E-03		<3.0E-03	
93-01371-C-2	W/dup	6.88E-04	19	4.95E+00	4			<1.2E-03		<5.7E-03		<2.8E-03	
Tank C-109, Co	ore Comp	osite. Fus	ion										
Core 47	•												
93-01358-H-1	F/smp	<2.5E-02		8.70E+02	4			<2.4E-01		<8.4E-01		<5.6E-01	
93-01358-H - 2				8.77E+02	4			<2.5E-01		<8.8E-01		<5.9E-01	
93-01358-H-3				1.75E-02	6			<5.4E-04		<1.1E-03		<5.3E-01	
Core 48				21762 62	·			VJ.4C-04		₹1.1E-03		<5.3E-04	
93-01363-H-1	F/smo	<2.4E-02		1.11E+03	- 4			<7.2E-02		<1.2E0		-7 CC 01	
93-01363-H-2				9.52E+02	4			<7.2E-02		<1.2E0		<7.5E-01	
Core 49	.,			0.022.02	•			17.3L-02		~1.1EU		<6.7E-01	
93-01371-H-1	F/smo	<1 2F-02		5.47E+02	4			3.90E-01	11	-E 2E 01		-2 FF 01	
93-01371-H-2				5.66E+02	4			3.33E-01	11	<5.3E-01		<3.5E-01	
	, dup	-1.5c 02		J.00L+02	4			3.33E-01	9	<5.1E-01		<3.5E-01	
Tank C-109, Ho	t Cell	(Results a	are in L	(ci/ml)									
93-01324-G-1		• •		4.18E-03	4	6.50E-05	20	8.68E-05	a	3 35 05		-25 AF	
93-01327-G-2				2.86E-03	4	5.67E-05	8	9.41E-05	9 4	3.3E-05	10	<2E-05	
93-01372-6-3				4.24E-05	4	<2E-06	o	<2E-06	4	2.1E-05	19	<1.5-E	
			•	7.276-03	*	~ C-UO		~2E-UD		<1E-06		<7E-07	

A=Acid Leach, W=Water Leach, F=Fusion, smp=sample, dup=duplicate, blk=blank * = Results are in μ Ci/ml.

Table 3-1b: SST Cores 47, 48, and 49, GEA Results, cont'd

Sample ID	*	Co(60) <i>µ</i> ci/g	+/- % error	Cs(137) <i>µ</i> ci/g	+/- % error	Eu(154) <i>µ</i> ci/g	+/- % error	Eu(155) <i>Ա</i> ci/g	+/- % error	Ат(241) <i>Џ</i> сі/g	+/- % error
Tank C-109, (Quarter	Segments,	Fusion								
Core 47-1B											
93-01355-H-1		<3.2E-02		3.17E+02	4	9.54E-01	6	8.82E-01	16	8.09E-01	20
93-01355-H-2		<2.3E-02		3.57E+02	4	8.10E-01	5	1.43E+00	11	6.92E-01	24
93-01355-H-3	F/OIK	<6.5E-04		3.80E-01	4	<1.3E-03		<2.4E-03		<1.3E-03	
Core 47-1C 93-01356-H-1	C/a==	42 2E 02		9 105.00							
93-01356-H-2		<2.2E-02		8.12E+02	4	<1.3E-01		<8.6E-01		<4.8E-01	
Core 47 1D	: / dup	~2.7L~UZ		7.31E+02	4	<1.3E-01		<8.6E-01		<4.8E-01	
93-01357-H-1	F/smn	<2.3E-02		9.71E+02	4	<1.2E-01		-1 150		.0.05.01	
93-01357-H-2				9.23E+02	4	<1.2E-01		<1.1E0 <9.4E-01		<6.0E-01	
Core 48-1C	.,			3.202,42	•	11.02 01		13.46-01		<5.1E-01	
93-01360-H-1	F/smp	<1.7E-02		1.17E+03	4	<7.7E-02		<1.0E0		<5.5E-01	
93-01360-H-2		<1.4E-02		1.14E+03	4	<7.4E-02		<1.2E0		<6.3E-01	
Core 48-10								1.220		-0.56-01	
93-01361-H-1	F/smp	<1.6E-02		1.22E+03	4	<9.0E-02		<1.2E0		<6.3E-01	
93-01361-H-2				1.11E+03	4	<1.0E-01		<1.2E0		<6.3E-01	
93-01361-H-3	F/blk	9.68E-04	15	1.31E+00	4	<7.0E-04		<3.8E-03		<1.8E-03	
Core 49-1B								-			
93-01365-H-1		<1.3E-02		1.21E+02	4	9.49E-01	4	1.19E+00	8	5.94E-01	18
93-01365-H-2	F/dup	<1.3E-02		1.15E+02	4	6.09E-01	4	6.67E-01	9	4.46E-01	15
Core 49-1C	_,										
93-01366-H-1		<1.1E-02		5.53E+02	4	<6.4E-02		<4.2E-01		<2.3E-01	
93-01366-H-2	F/dup	<1.6E-03		1.44E+02	4	<1.3E-02		<8.3E-02		<4.2E-02	
Core 49-10	-/										
93-01367-H-1		<1.1E-02		6.60E+02	4	<6.3E-02		<4.7E-01		<2.6E-01	
93-01367-H-2	r/ dup	<1.1E-02		7.43E+02	4	<6.1E-02		<5.1E-01		<2.7E-01	
Tank C-109, Q	uarter	Segment Ho	mogenizat	tion Test.	Acid						
Core 48-10			•								
93-01361-A-1T				8.52E+00							
93-01361-A-2T				1.66E+01							
93-01361-A-1B				8.81E+00							
				1 435+01							
93-01361-A-2B				1.43E+01							
93-01361-A-3	A/blk			8.73E-01							
93-01361-A-3 Core 49-1D				8.73E-01							
93-01361-A-3 Core 49-1D 93-01367-A-1T	A/smp			8.73E-01 3.54E+01							
93-01361-A-3 Core 49-1D 93-01367-A-1T 93-01367-A-2T	A/smp A/dup			8.73E-01 3.54E+01 4.34E+01							
93-01361-A-3 Core 49-1D 93-01367-A-1T 93-01367-A-2T 93-01367-A-1B	A/smp A/dup A/smp			8.73E-01 3.54E+01 4.34E+01 1.93E+01							
93-01361-A-3 Core 49-10 93-01367-A-1T 93-01367-A-2T 93-01367-A-1B 93-01367-A-2B	A/smp A/dup A/smp A/dup			8.73E-01 3.54E+01 4.34E+01 1.93E+01 2.74E+01							
93-01361-A-3 Core 49-10 93-01367-A-1T 93-01367-A-2T 93-01367-A-1B 93-01367-A-2B 93-01367-A-3	A/smp A/dup A/smp A/dup A/blk			8.73E-01 3.54E+01 4.34E+01 1.93E+01 2.74E+01 1.59E+00							
93-01361-A-3 Core 49-10 93-01367-A-1T 93-01367-A-2T 93-01367-A-1B 93-01367-A-2B 93-01367-A-3 Tank C-109, Co	A/smp A/dup A/smp A/dup A/blk	-48-49 Qua		8.73E-01 3.54E+01 4.34E+01 1.93E+01 2.74E+01 1.59E+00	nization	ı Test, Fus	ion	,			
93-01361-A-3 Core 49-1D 93-01367-A-1T 93-01367-A-2T 93-01367-A-1B 93-01367-A-2B 93-01367-A-3 Tank C-109, Co	A/smp A/dup A/smp A/dup A/blk	-48-49 Qua		8.73E-01 3.54E+01 4.34E+01 1.93E+01 2.74E+01 1.59E+00	nization	ı Test, Fus	ion				
93-01361-A-3 Core 49-1D 93-01367-A-1T 93-01367-A-2T 93-01367-A-1B 93-01367-A-2B 93-01367-A-3 Tank C-109, Co Core 49-1D 93-01367-H-1T	A/smp A/dup A/smp A/dup A/blk ores 47	-48-49 Qua		8.73E-01 3.54E+01 4.34E+01 1.93E+01 2.74E+01 1.59E+00 ment Homoge 7.13E+02	nization	ı Test, Fus	ion				
93-01361-A-3 Core 49-1D 93-01367-A-1T 93-01367-A-2T 93-01367-A-2B 93-01367-A-3 Tank C-109, Co Core 49-1D 93-01367-H-1T 93-01367-H-2T	A/smp A/dup A/smp A/dup A/blk ares 47 F/smp F/dup	-48-49 Qua		8.73E-01 3.54E+01 4.34E+01 1.93E+01 2.74E+01 1.59E+00 ment Homoge 7.13E+02 7.50E+02	nization	ı Test, Fus	ion				
93-01361-A-3 Core 49-1D 93-01367-A-1T 93-01367-A-2T 93-01367-A-1B 93-01367-A-2B 93-01367-A-3 Tank C-109, Co Core 49-1D 93-01367-H-1T	A/smp A/dup A/smp A/dup A/blk ores 47 F/smp F/dup F/smp	-48-49 Qua		8.73E-01 3.54E+01 4.34E+01 1.93E+01 2.74E+01 1.59E+00 ment Homoge 7.13E+02	nization	ı Test, Fus	ion				

A=Acid Leach, W=Water Leach, F=Fusion, smp=sample, dup=duplicate, blk=blank

Table 3-2: SST Cores 47, 48, and 49, Alpha Analysis Results

Sample ID	Tot Alpha #ci/g	+/- % error	Tot Alpha Pu μci/g	+/- % error	Am-241 <i>µ</i> ci/g	
Tank C-109 - Liquid Compos 93-01354-N-1 A/smp 93-01354-N-2 A/dup 93-01354-N-3 A/blk	< 5E-05 * < 5E-05 * < 1E-06 *		@ @ @		ଡ଼ ଡ ଜ	
93-03290-N	< 9E-07 *		@		0	
Tank C-109, Core Composite Core 47	, Water Leach					
93-01358-C-1 W/smp	5.08E-03	11				4
93-01358-C-2 W/dup	4.59E-03	12				
93-01358-C-3 W/blk	< 1E-04					
Core 48						•
93-01363-C-1 W/smp	1.37E-04	55				
93-01363-C-2 W/dup	1.32E-04	62				
93-01363-C-3 W/blk	1.522 04					
Core 49	6 015-04	17				
93-01371-C-1 W/smp	6.91E-04	18				
93-01371-C-2 W/dup	5.58E-04	10				
93-01371-C-3 W/blk						
Tank C-109, Core Composite Core 47	, Fusion					
93-01358-H-1 F/smp	9.24E-01	3	8.05E-01	7	2.50E-01	
93-01358-H-2 F/dup	1.06	3	9.49E-01	7	3.90E-01	
93-01358-H-3 F/blk	<3E-04	_	2.45E-04	19	<1E-04	
Core 48	-02 01					
93-01363-H-1 F/smp	5.79E-02	5	6.95E-02	7	8.88E-03	
93-01363-H-2 F/dup	7.12E-02	5	6.66E-02	7	1.13E-02	
•	7.126-02	3	0.006-02	,	1.136 02	
Core 49	1.22E-01	4	6.59E-02	7	1.31E-01	
93-01371-H-1 F/smp		4		7	1.35E-01	Ž.
93-01371-H-2 F/dup	1.36E-01	4	9.21E-02	,	96	•
STANDARD (% RECOVERY)			93			
BLANK			2.16E-06	13	<4E-07	
Tank C-109, Quarter Segmen	t Homogenizatio	n Test, A	cid			
Core 48	4 105 00					
93-01361-A-1T	6.18E-02	5				
93-01361-A-1B	6.74E-02	5				
93-01361-A-2T	5.00E-02	5				
93-01361-A-2B	5.26E-02	5				
93-01361-A-3	3.25E-04	42				
Core 49						
93-01367-A-1T	3.58E-02	6				
93-01367-A-1B	4.46E-02	5				
93-01367-A-2T	5.94E-02	5				
93-01367-A-2B	5.23E-02	5				
93-01367-A-3	4.52E-04	30				
33 01307 A 3	7.326 07	30				
Tank C-109, Hot Cell Blank						
93-01327-G-1 blk	1.2E-05 *	10				
93-01327-G-2 dup	1.0E-05 *	12				
93-01372-G-3 water	<2E-07 *					
	•					

A=Acid Leach, W=Water Leach, F=Fusion, smp=sample, dup=duplicate, blk=blank

[@] Total alpha concentration is so low that the analysis of alpha emitting isotopes was not performed. * Results are in μ ci/ml.

Table 3-3a: SST Cores 47, 48, and 49, Beta Analysis Results

Sample ID		Tot Beta [†] <i>µ</i> ci/g	+/- % error	\$r-90 <i>μ</i> ci/g	+/- % error	Tc-99 µci/g
Tank C-109 -	Liquid Comp	posite				
93-01354-N-1	A/smp	5.40E00 *	4	1.07E-02 *	11	1.54E-01 *
93-01354-N-2	A/dup	5.46E00 *	4	0.96E-02 *	11	1.58E-01 *
93-01354-N-3	A/blk	4.48E-05 *	30	<3E-05 *		<4E-06 *
93-03290-N		1.94E-04 *	8	1.74E-04	44	<3E-06 *
STANDARD (%)	RECOVERY)			100	7	106
BLANK	•			1.2E-04 *	79	<3E-06
Tank C-109, (Core Composi	te, Water Leach				
93-01358-C-1	W/smp	1.38E+01	3			
93-01358-C-2	W/dup	2.09E+01	3			
93-01358-C-3	W/blk	<6E-03	3			
Core 48	W/OIK	~0L ~03				
93-01363-C-1	W/smp	7.49E+00	3			
93-01363-C-2	W/dup	9.70E+00	3			
Core 49	w/ dup	3.702700	3			
93-01371-C-1	W/smp	9.04E+00	3			
93-01371-C-2	W/dup	8.43E+00	3			
33-013/1-6-2	w/ dup	0.432700	J			
Tank C-109, C Core 47	Core Composi	te, Fusion				
93-01358-H-1	F/smp	2.94E03	3	1.05E03	7	1.07E-01
93-01358-H-2	F/dup	2.55E03	3	1.30E03	7	1.09E-01
93-01358-H-3	F/blk	2.65E-02	3	<8E-03	·	<2E-03
Core 48						CL 40
93-01363-H-1	F/smp	1.41E03	3	1.90E02	8	1.17E-01
93-01363-H-2	F/dup	1.20E03	3	1.90E02	8	1.14E-01
Core 49	,				-	
93-01371-H-1	F/smp	2.42E03	3	8.77E02	7	9.36E-02
93-01371-H-2	F/dup	2.17E03	3	9.86E02	7	9.51E-02
STANDARD (% R	ECOVERY)			100		104
8LANK (μci	/ml)			1.2E-04 *		< 7E-6
Tank C-109 Ho	t Cell Blan	k				
93-01327-G-1	bik	3.08E-02 *	3			
93-01327-G-2	dup	3.27E-02 *	3			
93-01372-G	water	8.21E-05 *	4			

 $^{^{+}}$ Reported as Sr-90 - Y-90 A=Acid Leach, W=Water Leach, F=Fusion, smp=sample, dup=duplicate, blk=blank * Results are in μc i/ml.

Table 3-3b: SST Cores 47, 48, and 49, Beta Analysis Results, cont'd

Sample ID		Sr-90 µCi/g	+/- % error
Tank C-109, C	uarter Segments	, Fusion	
93-01355-H-1	F/smp	4.60E03	7
93-01355-H-2	F/dup	4.51E03	7
93-01355-H-3 Core 47-1C	F/blk	3.27E-02	25
93-01356-H-1	F/smp	4.56E02	,
93-01356-H-2	F/sinp F/dup	4.82E02	7 7
Core 47-1D	1 / Gup	4.02102	,
93-01357-H-1	F/smp	2.31E02	8
93-01357-H-2	F/dup	1.99E02	7
Core 48-1C	17 Odp	1.33602	,
93-01360-H-1	F/smp	1.59E02	7
93-01360-H-2	F/dup	1.44E02	8
STANDARD	(% RECOVERY)	96	J
BLANK	(µci/ml)	<2E-05	
	4		
Core 48-1D			
93-01361-H-I	F/smp	1.27E02	7
93-01361-H-2	F/dup	1.14E02	.7
93-01361-H-3	F/b1k	3.09E-01	9
Core 49-1B			
93-01365-H-I	F/smp	2.56E03	7
93-01365 - H-2	F/dup	2.23E03	7
93-01365-H-3	F/blk		
Core 49-1C			
93-01366-H-1	F/smp	2.02E02	7
93-01366-H-2	F/dup	1.89E02	7
93-01366-H-3	F/blk		
Core 49-1D			
93-01367-H-1	F/smp	1.88E02	7 7
93-01367-H-2	F/dup	1.97E02	7
93-01367-H-3	F/blk		
STANDARD	(% RECOVERY)	94	
BLANK	(μci/ml)	9.2E-05	

A=Acid Leach, W=Water Leach, F=Fusion, smp*sample, dup=duplicate, blk=blank

DATA REPORT PNL Analytical Chemistry tab. Radioanalytical Group, 325 Building

Procedure: LRB's:

WP #: M&TE:

Uranium Analysis Results -

Sample ID	Uranium mg/g	+/- % error
Tank C-109 - Drainable	: Liquid	
93-01354-N-L A/smp	3.65E-3 *´/	10
93-01354-N-2 A/dup	3.84E-3 *	10
93-01354-N-3 A/blk	< 2E-6 *	
93-03290-N	< 2E-6 *	
STANDARD % RECOVERY	98 /	
BLANK	< 7E-8 *	
Cores 47-48-49 Solid (Core Composite	
93-01358-H-L F/smp	1.17E+01	10
93-01358-II-2 F/dup	1.22E+01	10 -
93-01358-H-3 F/b1k	1.48E-02	10
93-01363-H-1 F/smp	3.00E+01	10
93-01363-H-2 F/dup	2.51E+01	10
93-01371-H-1 F/smp	7.63E+00	ĬŌ
93-01371-H-2 F/dup	7.42E+00	ĺŎ
STANDARD (% RECOVERY)	110	
BLANK	<5E-7	

A=Acid Leach, V=Vater Leach, F=Fusion, smp=sample, dup=duplicate, blk=blank * Results are in mg/mł.

Table 3-5: SST Cores 47, 48, and 49, Liquid Scintillation Counting Analysis Results

Sample ID		С-14 <i>µ</i> сі/m1	+/- % error	Н-3 <i>Д</i> сі/g		+/- % error	Se-79 <i>µ</i> ci/g
	- -						
	_iquid Composit						
	A/smp	2.5E-03		3.28E-03	*		
	A/dup	2.4E-03		3.29E-03	*		
93-01354-N-3	A/blk			<4E-07	*		
93-03290-N		2.7E-05		2.61E-05	*		
STANDARD (% R	ECOVERY)			94			•
Tank C-109, Co	ore Composite,	Water Leach					
Core 47							
93-01358-C-1		<5.0E-06		7.14E-03		3	
93-01358-C-2		6.3E-06		9.89E-03		3	
93-01358-C-3	W/blk	<5.0E-06		3.88E-03		3	
Core 48							
93-01363-C-1	W/smp	2.0E-05		6.15E-03		3	
93-01363-C-2	W/dup	1.6E-05		6.73E-03		3	
Core 49	•						
93-01371-C-1	W/smp	3.7E-05		7.23E-03		3	
93-01371-C-2		3.4E-05		5.47E-03		3	
STANDARD (% R				106			
BLANK	(μci/ml)			<5E-07			
Tank C-109, Co Core 47	ore Composite,	Fusion					
93-01358-H-1	F/smp						<8E-05
93-01358-H-2	F/dup						<8E-05
93-01358-H-3	F/blk						<8E-05
Core 48							
93-01363-H-1	F/smp						6E-05
93-01363-H-2	F/dup						<5E-05
Core 49	•						
93-01371-H-1	F/smp						<5E-05
93-01371-H-2	F/dup						5E-05
BLANK (Mci.	/ml) `						<2E-07 *
•							

A=Acid Leach, W=Water Leach, F=Fusion, smp=sample, dup=duplicate, blk=blank * Results are in μ ci/ml.

Table 3-6: SST Cores 47, 48, and 49, Pu MS Isotopic Percent

Sample ID		234	235	236	238	
Tank C-109 - 93-01354-N-1 93-01354-N-2 93-01354-N-3 93-03290-N		U conce	entration t	oo low for Mas	s Spec ana	lysis
	ore Composite, Fusi	on				
Core 47 93-01358-H-1	5 /	0.0050	0.5500	0.0100		
93-01358-H-2	F/smp F/dup	0.0059 0.0062	0.6580	0.0100	99.3261	
93-01358-H-3	F/blk	0.0062	0.6566	0.0107	99.3265	
Core 48	r/DIK					
93-01363-H-1	F/smp	0.0056	0.682	0.0059	99.3065	
93-01363-H-2	F/dup	0.0058	0.6883	0.0049	99.3010	
Core 49	,			2,00		
93-01371-H-1	F/smp	0.0051	0.6792	0.0079	99.3077	
93-01371-H-2	F/dup	0.0056	0.6713	0.0089	99.3141	
Sample ID		238	239 	240	241	242
Tank C-109 -	Liquid Composite					
93-01354-N-1						
93-01354-N-2	•	concentra	tion too 1	ow for Mass Sp	ec analysis	•
93-01354-N-3	A/b1k				•	
93-03290-N						
Tank C-109, Core 47	ore Composite, Fusi	on				
93-01358-H-1	F/smp	0.005	93.3295	6.5200	0.1214	0.0239
93-01358-H-2	F/dup	0.005	93.1179	6.7311	0.1217	0.0242
93-01358-H-3	F/blk ~					0.0676
Core 48						
93-01363-H-1	F/smp	0.005	97.8337	2.1208	0.029	0.0113
93-01363-H-2	F/dup	0.017	97.4374	2.4782	0.0438	0.0239
Core 49					•	
93-01371-H-1	F/smp	0.011	94.9880	4.8456	0.1212	0.0346
93-01371-H-2	F/dup	0.017	94.9378	4.9115	0.1025	0.0311

A=Acid Leach, W=Water Leach, F=Fusion, smp=sample, dup=duplicate, blk=blank

Extractable Organic Halide Analysis Results

The Core 49 core composite (93-01371) was analyzed in duplicate for the presence of extractable organic halides. Insufficient sample was available from the Core 47 and 48 composites to perform this analysis. Three aliquots of the sample were extracted in the SAL according to procedure PNL-ALO-320, "Extractable Organic Halides." These samples (a blank, sample, sample duplicate, matrix spike) were then analyzed using method PNL-ALO-320 using a Dohrmann, microcoulometric titration, halogen analyzer.

Table 4-1: SST Core 49, Extractable Organic Halide Data

ACL Sample Number	<u>Description</u>	<u> Halide Conc.</u>
93-01371-F1	CORE-49 SAMPLE	ll μg/g
93-01371-F2	CORE-49 SAMPLE DUPE	<10 µg/g
93-01371-F3	METHOD BLANK	<10 μg/g
93-01371-F4	CORE-49 MATRIX SPIKE	91% Recovery

The Halide concentrations are based on the total wet weight of the sample aliquot received from the hot cells.

Each aliquot from Sample 93-01371 appeared to be similar in color and consistency. The color of the sample was tan and appeared very dry. A pH adjustment was required prior to extraction. Halide was detected at the detection limit for this method, which is $10~\mu g/g$. The spike recovery was within the current established guidelines for this procedure.



Pacific Northwest Laboratories Battelle Boulevard P.O. Box 999 Richland, Washington 99352 Telephone (509)

376-2639

September 9, 1993

Brett C. Simpson, R2-12 Westinghouse Hanford Company P. O. Box 1970 Richland, WA 99352

Dear Brett:

ACL RESPONSE TO THE C-109 DATA VALIDATION REPORT

Please find attached the Pacific Northwest Laboratory Analytical Chemistry Laboratory response to the HASM validation report of the Tank C-109 data. This response was originally issued to K. N. Pool (HASM). The content of this document is only a response to the issues raised in the validation report and in no way does our response requalify the data. We have asked in this document that the usability of the radiochemical data be reevaluated in terms of its adequacy for its intended use.

Please feel free to call if you have questions regarding the C-109 data or the attached document.

Sincerely,

Susan G. McKinley

PNL TWC Project Manager

Analytical Chemistry Laboratory

SGM/rmn

Attachment

cc: K. J. Kuhl-Klinger Project File - 16021 File/LB

ACL RESPONSE TO C-109 DATA VALIDATION REPORT

The following response is based upon the Chemical and Radiochemical Data Validation Narrative portions of the C-109 Data Validation Report. This response is formatted in the exact order as presented in the Report.

Inductively Coupled Argon Plasma (ICP)

Regarding IEC Corrections: The application of qualifiers to IEC corrected data had minimal impact on this data package. Therefore, no response is warranted.

Arsenic by ICP: The Validator qualified arsenic results as "R" (unreliable) because of unacceptable initial calibration verification (ICV) results. Please note that the ICV does not contain arsenic; the "true" concentration reported was actually that for barium, not arsenic. The arsenic is compared using a "MCV" standard at 10 ppm. A "Don't Say It--Write It" (DSI) to file is attached to this response for inclusion in front of the ICP raw data. This DSI alerts reviewers to the error. It should be noted that arsenic was reported from the GFAA analysis as well. Since GFAA is more sensitive than ICP for arsenic, the GFAA data should be used for assessment.

No additional issues of significance were noted for the remaining inorganic analyses.

Radiochemical Data Validation

The Validator qualified most radiochemical data as "R" (unreliable) due to lack of evidence of spikes, carriers, or tracers.

<u>Total Alpha/Beta</u>: The Validator references a requirement for matrix spikes in WHC-EP-210; the laboratory does not follow this plan. The ACL follows the Technical Project Plan (TPP) and Quality Assurance Project Plan (QAPjP). However, the qualification of data as "unreliable" due to lack of a matrix spike appears extreme; no chemical separations are performed, and alpha and beta can be compared against the major contributing isotopes. The sample is evaporated onto a counting plate and total count rate is reported. The Validator references the lack of Counter Control data. Counter Control data start on page E04-204 for alpha and E05-174 for beta; this control data is used as the daily instrument performance check and confirms validity of the original calibration. Initial calibration data is not a required submission under this project; counter control data is a required submission.

Americium-241: Matrix spikes, tracers or carriers were not performed and the data was qualified as "unreliable." A standard and blank were processed similarly to the samples. This standard recovered at 96%, indicating that no processing errors occurred. Plutonium and americium-241 account for the major alpha present, and the sum of these isotopes compares well with the total alpha. The ACL does not believe qualification of the data as "unreliable" is justified.

<u>Total Plutonium</u>: Matrix spikes, tracers or carriers were not performed and the data was qualified as "unreliable." As noted above, total plutonium and

americium generally account for most of the alpha activity, and this data compares well with the total alpha measurements. The ACL does not believe qualification of the data as "unreliable" is justified.

Neptunium-237: Tracers were not run and the data was therefore qualified as "unreliable." A standard prepared similarly to the samples recovered well, indicating adequate sample processing technique. The low levels of neptunium-237 detected appear reasonable when added to plutonium and americium for comparison to the total alpha. Qualification of the low-level neptunium-237 does not appear to be reasonable.

Strontium-90: Carriers were not run and the data was therefore qualified as "unreliable." Good agreement was noted between the sum of the strontium-90/yttrium-90 and cesium-137 with the total beta, three independent techniques. For this reason, the ACL does not believe qualification of results as "unreliable" is justified.

<u>Technicium-99</u>: Spikes or carriers were not performed and the data was therefore qualified as "unreliable." The laboratory has not found a suitable tracer for routine use. Standards processed similarly to the samples recovered well, indicating acceptable sample processing. The technicium-99 and strontium-90 together compare well with the total beta. Qualification of the results as "unreliable" does not appear to be justified.

<u>Carbon-14</u>: Matrix spikes were not performed and the data was therefore qualified as "unreliable." The National Institute of Standards and Technology (NIST) standards are prepared and analyzed similarly to the samples. Recoveries were good, indicating acceptable preparation and analysis. Qualification of the results as "unreliable" does not appear to be justified.

<u>Selenium-79</u>: A carrier was not run and the data was therefore qualified as "unreliable." This statement is incorrect. The ACL adds metal carrier to every sample and determines the yield gravimetrically. No data should have been qualified as "unreliable."

<u>Total Uranium</u>: No problems were noted. No response required.

Gamma Energy Analysis (GEA): No problems were noted. No response required.

<u>Mass Spectrometry</u>: All data was qualified as "unreliable" because of missing data for daily standard run. A standard is run daily, when in use, for this technique. See pages E09-026 through E09-034 for daily standards.

Physical Testing: No problems were noted. No response required.

Additional Concerns: The Validator noted that the radiochemical calculations were extremely difficult to verify because units were not included. The ACL takes exception to the Validator's comment. The raw data indicates counts, time in minutes and all sample processing factors. The only units missing are d/c or c/d for the efficiency; d/c or c/d can be ascertained quite easily by noting whether the efficiency is greater or less than 1.0.

ACL SUMMARY

The ACL is most concerned by the "unreliable" qualification of the radiochemical data. Although carriers, tracers and/or spikes were not performed for the majority of isotopes tested, the agreement between independent techniques should be supportive of the reliability of results. Consideration should also be given to the fact that standards are processed similarly to the samples after hot-cell preparation. Standard recoveries were acceptable for all isotopes reported, indicating acceptable sample processing technique. If one assumes acceptable sample processing technique, then matrix problems would be the final area of concern. Again, the fact that comparison of results across multiple, independent techniques showed consistency in agreement should further support data reliability. The ACL would therefore ask that usability of the radiochemical data be reevaluated in terms of adequacy for its intended use.

It is not our intent to question whether tracers, carriers and/or spikes would have given more credence to the data, but rather that these quality control checks were not performed based upon verbal agreements between Westinghouse and the ACL at the time. This data package is consistent with previous submissions; until your validation report was received, there was no indication that a problem existed. It has been the ACL's perception that previous data were found to be adequate for the intended use.

This page intentionally left blank.

DISTRIBUTION

Number of Copies

<u>OFFSITE</u>

25	U.S. Department of Energy EM-35, Trevion II Washington, D.C. 20585
	John C. Tseng
1	U.S. Department of Energy Savannah River Operations Office P.O. Box A Aiken, South Carolina 29808
	Thomas C. Temple
1	Charles S. Abrams 1987 Virginia Idaho Falls, ID 83404
1	David O. Campbell 102 Windham Road Oak Ridge, TN 37830
1	Fred N. Carlson 6965 North 5th West Idaho Falls, ID 83401
1	Donald T. Oakley 409 12th Street SW, Suite 310 Washington, DC 20024-2188
1	Arlin K. Postma 3640 Ballard Road Dallis, Oregon 97338
1	William R. Prindle 1556 Crestline Drive Santa Barbara, CA 93105
1	Alfred Schneider 5005 Hidden Branches Drive Dunwoody, GA 30338

Number of Copies

OFFSITE

2	Argonne National Laboratory 9700 South Cass Avenue CMT/205 Argonne, IL 60439-4837
	James C. Cunnane Theodore R. Krouse
1	Air Products & Chemicals, Inc. 7201 Hamilton Blvd Allentown, PA 18195-1501
	George E. Schmauch
1	Battelle Columbus Laboratories 505 King Avenue Columbus, OH 43201-2693
	James A. Gieseke
2	Brookhaven National Laboratory Upton, NY 11973
	Kamal K. Bandyopadhyay Morris Reich
1	<u>Design Science, Inc.</u> 163 Witherow Road Sewickley, PA 15143
	Gary Powers
1	Fauske and Associates, Inc. 16W070 W. 83rd St. Burr Ridge, IL 60521
	Hans K. Fauske
1	Florida State University Department of Chemistry B-164 Tallahassee, FL 32306
_	Greg R. Choppin

Number of Copies

<u>OFFSITE</u>	
1	<u>Harvard University</u> 295 Upland Avenue Newton Highlands, MA 02161
	Melvin W. First
1	<u>Hazards Research Corporation</u> 200 Valley Road, Suite 301 Mt. Arlington, NJ 07856
	Chester Grelecki
1	Lawrence Livermore National Laboratory P.O. Box 808, L-221 Livermore, CA 94550
	Billy C. Hudson
4	Los Alamos National Laboratory P.O. Box 1663 Los Alamos, NM 87545
. *	Steve F. Agnew Steve W. Eisenhawer Thomas E. Larson L. Harold Sullivan
1	MIT/Department of Nuclear Engineering 77 Massachusetts Ave. Room 24-102 Cambridge, MA 02139
	Mujid S. Kazimi
1	Nuclear Consulting Services, Inc. P.O. Box 29151 Columbus, OH 43229

J. Louis Kovach

Number of Copies

OFFSITE

3	Oak Ridge National Laboratory
	Emory D. Collins
	P.O. Box 2008
	7930, MS-6385
	Oak Ridge, TN 37831-6385

Charles W. Forsberg P.O. Box 2008 MS-6495 Oak Ridge, TN 37831-6495

Thomas S. Kress P.O. Box 2009 9108, MS-8088 Oak Ridge, TN 37831-8088

Rice University
5211 Paisley
Houston, TX 77096

Andrew S. Veletsos

Sandia National Laboratory
P.O. Box 5800
Albuquerque, NM 87185

Scott E. Slezak

Science Applications International Corporation
12850 Middlebrook Road
Trevion I, Suite 300
Germantown, MD 20874

Ray S. Daniels (3) John M. Saveland

1 University of South Carolina
Department of Electrical and Computer Engineering
Swearingen Engineering Center
Columbia, SC 29208

Joseph S. Byrd

Number of Copies

<u>lumber of Copies</u>		
<u>OFFSITE</u>	•	
1	University of Washington Center for Process Analytical Chemistry Chemistry Department BG-10 Seattle, WA 98195	
	Bruce R. Kowalski	
1	Vanderbilt University P.O. Box 1596, Station B Nashville, TN 37235	
	Frank L. Parker	
	•	
<u>ONSITE</u>		
18	U.S. Department of Energy. Richland Field Office	
	R. F. Christensen (8) J. M. Clark (3) R. E. Gerton A. G. Krasopoulos T. Noble R. O. Puthoff Public Reading Room RL Docket File (2)	R3-72 R3-72 R3-72 A4-81 R3-72 A5-10 A1-65 A3-11
12	Pacific Northwest Laboratory	
-	R. T. Allemann S. A. Bryan N. G. Colton B. M. Johnson M. A. Lilga S. G. McKinley K. H. Pool R. D. Scheele G. F. Schiefelbein D. M. Strachan J. M. Tingey PNL Technical Files	K7-15 P7-25 P8-30 K1-78 P8-38 P7-22 P8-44 P7-25 P8-38 K2-38 K2-44 K1-11

Number of Copies

<u>ONSITE</u>

104 <u>Westinghouse Hanford Company</u>

W.	Τ.	Alumkal	R2-52
Н.	Bal	oad .	R2-78
D.	В.	Bechtold	T6-09
J.	R.	Bel1	R3-09
J.	В.	Billetdeaux	R2-08
Ř.	J.	Blanchard	R1-17
	Č.	Board	S1-57
	Ĺ.	Borsheim	R2-11
	Ċ.	Boyles	R1-49
	Ğ.	Burk	B3-25
	Ĵ.	Cash (5)	R2-78
M.	Ď.	Crippen	L5-31
	Μ.	Christensen	H4-21
Ŕ.	D.	Crowe	H4-68
	J.	Dechter	R2-54
Č.		Figh-Price	R2-31
Ğ.		Dukelow (2)	R2-78
	Ĺ.	Dunford	R1-51
	j.	Forbes	R1-08
	D.	Fowler	R2-11
	Ĺ.	Fox	L5-01
	T.	Frater	R1-51
	C.	Fulton	R2-31
	Α.	Gasper	R2-08
S.	D.	Godfrey	R1-51
J.	M.	Grigsby	H4-62
T.	W.	Halverson	R2-54
D.	G.	Hamrick	S6-15
Н.	D.	Harmon	R2-52
	Μ.	Held	R3-09
J.	Μ.	Henderson	S4-55
D.	L.	Herting	T6-09
J.	D.	Hopkins	R2~11
R.	D.	House	R1-05
M.	W.	Howard	G1-75
M.	N.	Islam	R3-08
D.	W.	Jeppson	L5-31
J.		Jewett	T6-09
D.	R.		R2-08
	W.		R2-11
	L.		H0-34
C.	Α.	Kuhlman	B3-30
		nmerer	H4-62
		Lenseigne	R2-75

Number of Copies

ONSITE

Westinghouse Hanford Company (continued)

D. A. Marsh	A3-05
J. D. McCormack	L5-31
N. G. McDuffie	R2-78
J. M. McLaren	H0-34
J. E. Meacham	R2-78
J. E. Meacham S. J. Mech	L5-55
J. P. Menard	R2-40
J. W. Neskas	R2-11
N. J. Milliken	H4-63
A. F. Noonan	R2-12
P. C. Ohl	R1-30
M. A. Payne	R2-50
R. S. Popielarczyk	R1-30
D. N. Price	R2-14
J. G. Propson	R2-18
R. E. Raymond	R2-54
I. E. Reep	R2-08
F. R. Reich	L5-63
E. L. Renner	R2-08
D. A. Reynolds	R2-11
E. L. Renner D. A. Reynolds J. H. Roecker C. C. Scaief C. P. Schroeder M. H. Shannon	B1-59
C. C. Scaief	L7-06
C. P. Schroeder	L7-06
M. H. Shannon	H4-61
N. L. Simon	R2-78
B. C. Simpson (10) S. R. Tifft	R2-12
S. R. Tifft	H6-26
H. Toffer	H0-38
J. D. Thomson R. E. Vandercook	R1-30
R. E. Vandercook	S6-17
W. T. Watson	H0-38
R. K. Welty	R1-80
W. P. Whiting	B3-25
J. C. Wiborg	H4-60
W. D. Winkelman	L5-55
W. I. Winters	T6-50
D. D. Wodrich	R2-31
D. D. Wodrich	H0-30
W. F. Zuroff	R2-14
Central Files	L8-04
Document Processing and Distribution (2)	L8-15
EDMC	H6-08
Information Release Administration (3)	R1-08
TFIC	R1-20

This page intentionally left blank.

Number of Copies

ONSITE

Westinghouse Hanford Company (continued)

D. A. Marsh	A3-05
J. D. McCormack	L5-31
N. G. McDuffie	R2-78
J. M. McLaren	H0-34
J. E. Meacham	R2-78
S. J. Mech	L5-55 R2-40
J. P. Menard J. W. Neskas	R2-40
N. J. Milliken	H4-63
A. F. Noonan	R2-12
P. C. Oh1	R1-30
M. A. Payne	R2-50
R. S. Popielarczyk	R1-30
D. N. Price	R2-14
J. G. Propson	R2-18
R. E. Raymond	R2-54
I. E. Reep	R2-08
F. R. Reich	L5-63
E. L. Renner	R2-08 R2-11
D. A. Reynolds J. H. Roecker	B1-59
J. H. Roecker C. C. Scaief	L7-06
C. P. Schroeder	L7-06
M. H. Shannon	H4-61
N. L. Simon	R2-78
B. C. Simpson (10)	R2-12
S. R. Tifft	H6-26
H. Toffer	H0-38
J. D. Thomson	R1-30
R. E. Vandercook	S6-17
W. T. Watson	H0-38
R. K. Welty	R1-80
W. P. Whiting	B3-25
J. C. Wiborg	H4-60
W. D. Winkelman	L5-55
W. I. Winters	T6-50
D. D. Wodrich	R2-31
D. D. Wodrich W. F. Zuroff	H0-30
Central Files	R2-14 L8-04
Document Processing and Distribution (2)	L8-15
EDMC	H6-08
Information Release Administration (3)	R1-08
TFIC	R1-20

THIS PAGE INTENTIONALLY LEFT BLANK